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Hydrophobic soil :

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HYDROPHOBIC SOIL:
A LOW - COST ALTERNATIVE
TO CLAY LINING MATERIALS

by

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A Thesis
Presented to the Graduate Committee
of Lehigh University
in Candidacy for the Degree of
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CERTIFICATE OF APPROVAL

This thesis is accepted and approved in partial fulfillment of the requirements for the degree of Master of Science in Civil Engineering.

May 18, 1988
Date

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Department Chairman

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TABLE OF CONTENTS

ABSTRACT

Failures in practice of several clay lining systems to retain hazardous or toxic wastes have resulted in intensified efforts to improve upon or find alternatives to such materials. Most of the work has focused upon the concept of physical impedment of the fluid by the use of fine-grained substances which contain minute pore openings. This investigation, however, considers the concept of water repellence, or the actual repulsion of fluid by the soil particles. Such a phenomenon is related to the surface tension that exists between the solid surface and the fluid and is measured by the liquid-solid contact angle. Research conducted upon naturally-occurring water repellent or hydrophobic soils have indicated that an organic coating, specifically a humic substance, covers the soil particles and significantly alters their surface characteristics.

A particular advantage of this approach must be its relative low cost. With this in mind, a plentiful, inexpensive source of humic substances (activated sludge) is chosen to mix with a random sample of sandy soil to create a coated material. This new material is then heated at specified durations and temperatures to intensify the hydrophobic effect. Subsequent tests reveal

a high degree of water repellence in the treated and heated soils. Specifically, water drop penetration times increase from instantaneous to 540 seconds for the 200°C/ 5 minute sample. Also, the liquid-solid contact angle increases from 58.1° to 77.9° and the rate of permeability decreases from 2.15×10^{-2} cm/sec to 4.43×10^{-4} cm/sec. While this is a significant improvement, it is not enough to replace a clay lining material. In addition, the problem of soluability of the organic coating must yet be solved. Nevertheless, the concept of artificially-produced hydrophobic soil is proven to be feasible and worthy of further investigation.

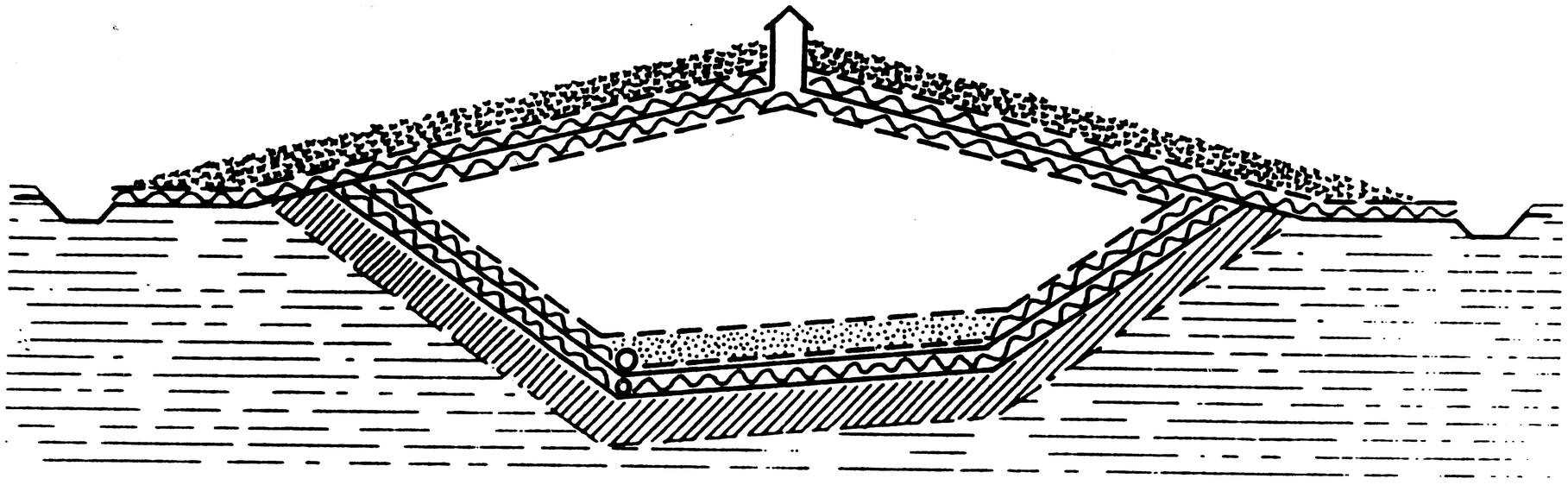
I. INTRODUCTION

The permeability or hydraulic conductivity with respect to a particular permeant (usually water) is a basic engineering characteristic of any given soil mass. Specifically, it describes the rate at which a certain fluid passes through the soil fabric. Such a property is of special interest whenever groundwater flow is to be considered. These design situations include earth dams, aquifer discharge or recharge (water supply), construction dewatering, contaminant transport, and reservoir lining systems. Permeability is primarily a function of such physical factors as particle size and distribution, porosity, and the shape and arrangement of the particles (Todd, 1980). For the most part, though, permeability is directly proportional to particle size and, therefore, the size of the pore openings. As a result, sands and gravels have a higher permeability than silts and clays.

Over the last several years, one specific area of concern has taken advantage of this fact. The use of low-permeability soils, especially clays, has become an attractive solution to the problem of waste containment. Public awareness of this situation has recently grown, primarily because of the increased severity of the wastes

requiring disposal (hazardous or toxic) and the lack of available space to put them. As a result, landfill design criteria have been vastly improved and new alternative technologies have been developed. With regard to landfills, the improved criteria includes the design of composite lining systems (see Figure 1). These involve the use of both a low permeability clay soil and a geomembrane with associated geotextiles (Giroud, 1984). The effectiveness, therefore, of such a lining system depends in part of the ability of a highly engineered clay material (usually bentonite) to retard the flow of contaminated permeants.

Unfortunately, most early laboratory permeability tests were made under sterile conditions - pure clay materials and clean water. The results of these investigations were used in the design of landfill lining systems. Today, however, some of these same systems are leaking with permeability rates several orders of magnitude higher than predicted in the lab (Dunn, 1986). It has been found that the construction techniques as well as the pore fluids themselves have a profound influence over clay behavior (Fang, 1986). As a result, it is now known that a thorough understanding of the clay-pore fluid interaction is required before the longterm behavior of these systems may be predicted. In



COVERING MATERIAL

CLAY

SAND / GRAVEL

SUBGRADE

GEOMEMBRANE

GEOTEXTILE

GEONET

DRAINING PIPES

Figure 1. Composite landfill lining system including clay and geosynthetics.

many cases, this may render the clay liner prohibitively expensive if not physically impractical.

Naturally, these problems have provided the impetus for research into low-cost alternatives such as fly ash admixtures (Soliman et al., 1986). In general, these attempts have relied on fine grain sizes and minute pore openings to provide adequate permeability rates. The concept of hydrophobic soil, however, takes a completely different approach. As its name implies, this soil actually repels water. In addition, this phenomenon has absolutely nothing to do with smaller grain sizes or pore openings. In fact, the opposite is true. It has been found that the degree of water repellency is directly proportional to grain size. It is reasonable to believe that this fact alone will eliminate some of the problems associated with clays.

Hydrophobic soil has been found to occur in nature. This investigation has examined the causes of this unique occurrence to determine its reproducibility. The intention is to establish a feasible method to artificially induce hydrophobic behavior in soils. Permeability, water drop penetration, and liquid-solid contact angle tests will be used to ultimately determine the success of the procedure. In an effort to provide a low-cost solution, a random granular soil is chosen to

represent a typical on-site material. The treatment procedure is purposely kept simple and inexpensive to simulate a low-cost, practical field application. It is believed, therefore, that the value of this approach lies in its simplicity and practicality.

II. REVIEW AND BACKGROUND

Early Observations

The problem of soil wettability is one that has received significant attention in the recent past. The agriculture industry has maintained a particular interest as it is an important aspect of an efficient irrigation system. Obviously, the effectiveness of any plant growth is highly dependent upon the amount of water that reaches its roots. Therefore, any degree of water repellency exhibited by the soil would be a detriment. An early observation of such a phenomenon (Jamison, 1947) indicated that the surface soil beneath Florida citrus trees displayed a marked resistance to wetting. Figure 2 clearly shows that the repellency was most noticeable under the trees within the leaf drip line from a depth of about 1 to 12 inches. It was surmized at the time that this condition was the result of a coating on the soil particles of insoluble calcium and magnesium soaps (Wander, 1949) and could be treated by a combination of stirring and wetting the soil.

Another major industry that has been affected by the problem of soil wettability is that of forestry or land management. In particular, the soils of many regions in the western United States have been observed to exhibit naturally occurring water repellency to some

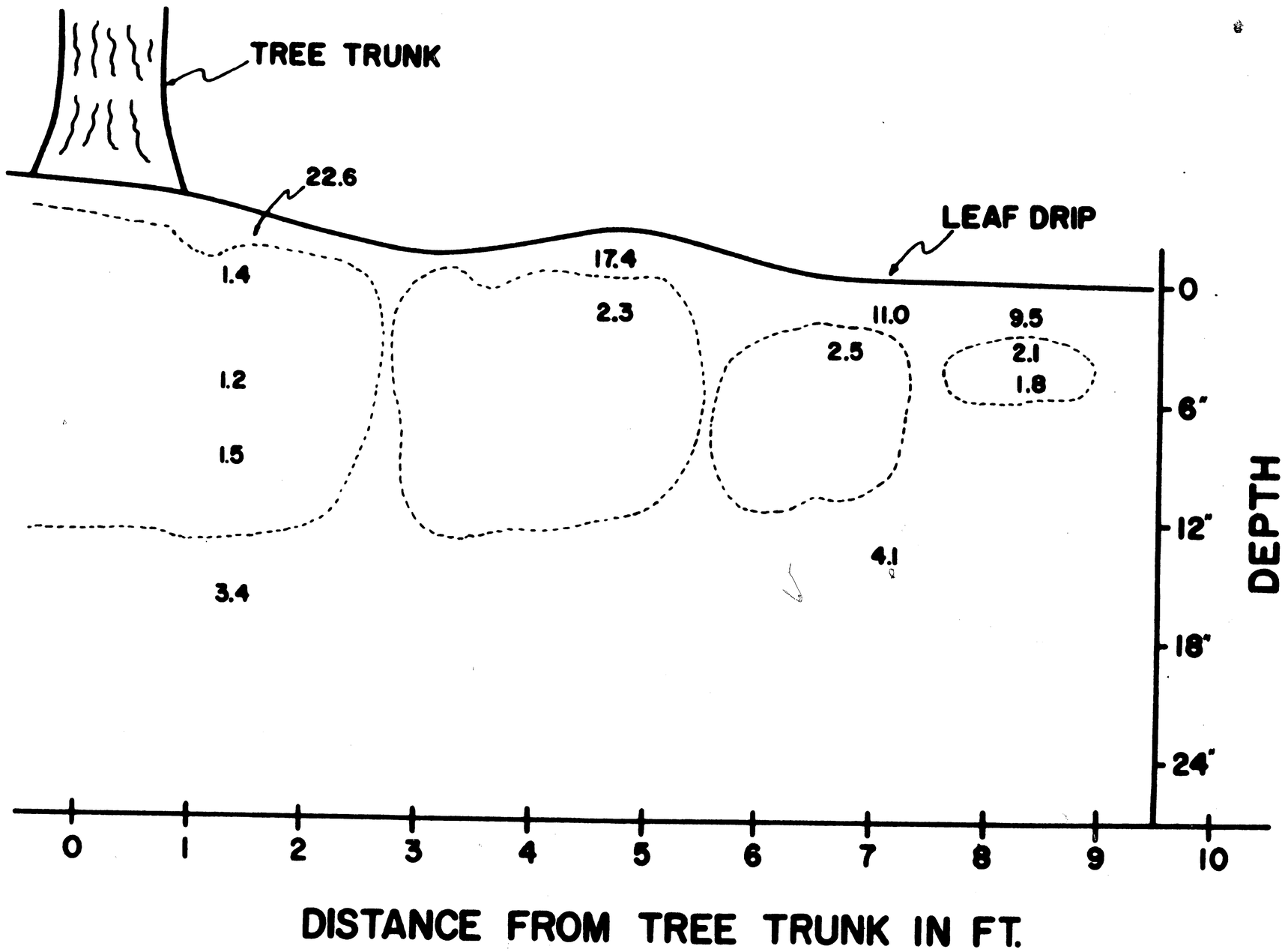


Figure 2. A vertical cross section of moisture under a citrus tree. Moisture contents at various depths are indicated. (After Jamison, 1947)

extent (Krammes and DeBano, 1965; DeBano, 1969c). This situation has resulted in extremely high runoff rates and debris yields, which in turn greatly affects flood control. It was observed that the degree of water repellency is greatly enchanced after the soil had been exposed to wildfire temperatures (DeBano and Krammes, 1966). These studies offered similar conclusions - the phenomenon of water repellency in soil results from an organic coating likely originating from the litter of the native brush-like plants (chaparral). Furthermore, heat treatment can increase the condition appreciably. However, too much heat will destroy it altogether. Figure 3 indicates that this is a function of both heating temperature and duration.

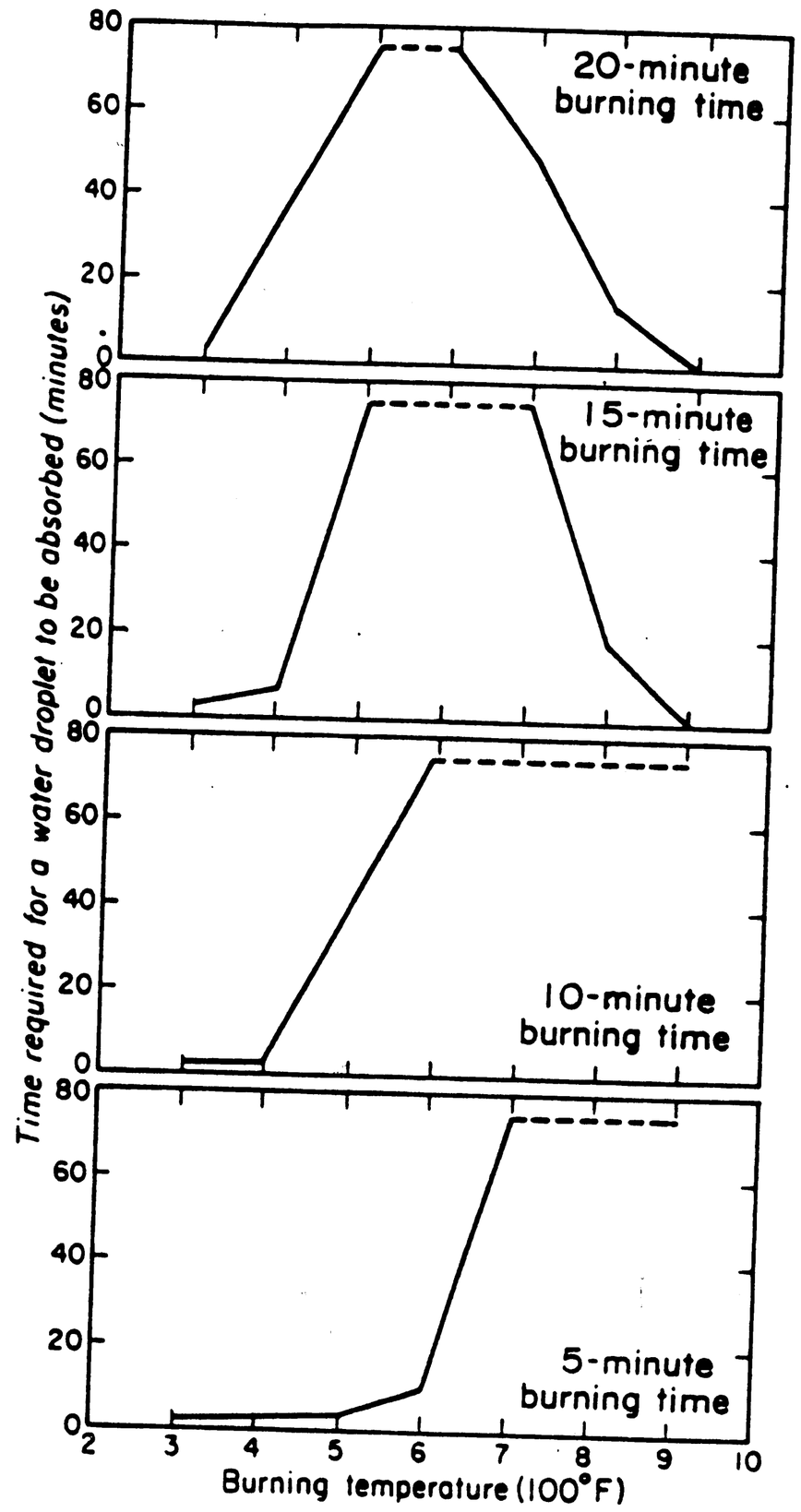


Figure 3. The relationship between soil non-wettability and temperature at four burning times. The broken line indicates the virtually impenetrable condition where the time for the water droplet exceeded 75 minutes. (After DeBano and Krammes, 1966)

Characterization Techniques

Early research focused on appropriate methods of characterizing water repellency. Specifically, Water Drop Penetration Time, critical surface tension, and the liquid-solid contact angle were defined (Letey, 1969). The most meaningful (and difficult to measure) of these is the liquid-solid contact angle, or wetting angle. The affinity or repellency of a solid surface for water relates to the attractive forces between them. If this attraction is greater than that between the individual water molecules, the water will spread out on the solid surface. If this attraction is less, the water will be repelled and will "ball up", forming an angle ($\theta > 0^{o}$) between the liquid-air and the liquid-solid interfaces (DeBano, 1969a). Figure 4 shows the mechanical equilibrium as expressed by:

$$T_{sv} - T_{sl} = T_{lv} \cos \theta \qquad (1)$$

where:

T_{sv} = tension at solid-vapor interface
T_{sl} = tension at solid-liquid interface
T_{lv} = tension at liquid-vapor interface
θ = liquid-solid contact angle

In soils, the liquid-solid contact angle is the effective angle formed by a water meniscus in contact with the soil pore walls. If the angle is greater than 90^{o}, water will

$$T_{sv} - T_{sl} = T_{lv} \cos \Theta$$

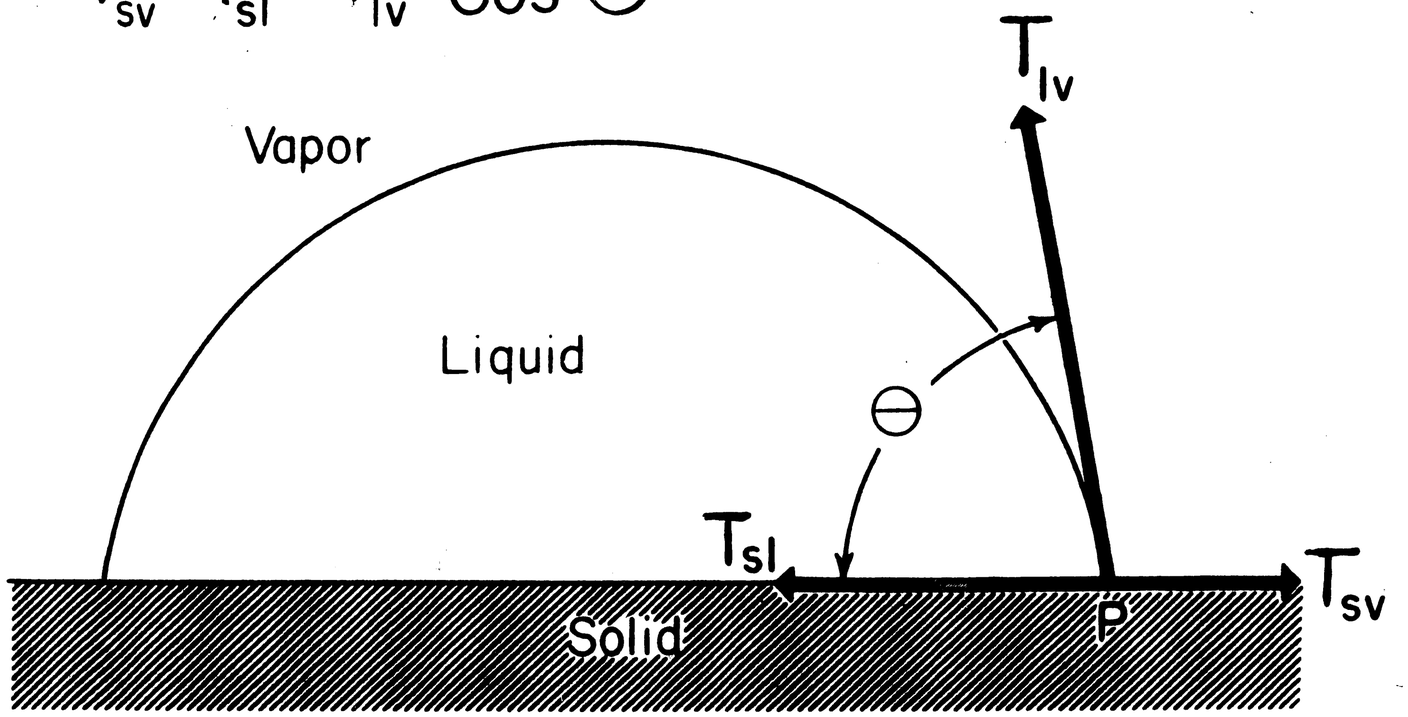


Figure 4. A water droplet at mechanical equilibrium on a solid surface. The angle formed between the liquid surface and the solid surface at the point of contact is the liquid-solid contact angle. (After DeBano, 1969a)

not penetrate or move through the soil fabric. If the angle is between 90^{o} and 0^{o}, penetration time will vary from infinite to instantaneous. Measurement of this parameter will be discussed in a subsequent section.

An important conclusion obtained from this early research is that water repellency is not a function of pore size (Letey et al., 1962a). Instead, water repellency is directly related to the surface tension between the solid and the liquid. Also, it is important to note that the liquid-solid contact angle is not necessarily constant, especially for soil. It is possible that the hydrophobic coatings may dissolve over time, thereby changing the properties of the solid surface. Finally, penetration may be affected by other factors, including gravity, which may alter the apparent value of the angle (Letey et al., 1962b).

While the liquid-solid contact angle is the best physical characterization of the hydrophobic condition, the measurement of this angle is not convenient when a large number of samples are to be tested, the sample sizes are small, or a field survey is to be conducted. With this in mind, a much simpler parameter was developed known as the Water Drop Penetration Time (WDPT) (Savage et al., 1969; Watson and Letey, 1970). A drop of water is simply placed upon the surface of the soil. If the

drop of water "balls up", the liquid-solid contact angle is greater than 90^{o}. The test, then, measures the time taken by the drop to penetrate the sample. For most water repellent soils, this may take seconds or minutes. Since a drop will only penetrate a porous medium once the contact angle is less than 90^{o}, the angle does not remain constant with time. The change is a result of falling surface tension values brought about by solid-liquid interaction. Nevertheless, the Water Drop Penetration Time gives a good indication of initial conditions. In this way the test may be used for relative determination of the extent of hydrophobic behavior in a variety of samples. Obviously, the longer the Water Drop Penetration Time, the more water repellent the sample.

The preceeding method, in all of its simplicity, was found to be inaccurate at both extremes of the Water Drop Penetration Time (Watson and Letey, 1970). At very long durations, the test becomes impractical. At very short durations (less than 2 seconds), it becomes difficult to distinguish between samples. Since the liquid-solid contact angle is dependent upon the original surface tension of the applied liquid, measuring the critical surface tension becomes a more useful characterization technique. A small period of time is arbitrarily chosen (i.e. 25 seconds) at which the surface tension is

assumed to be associated with a 90^{o} contact angle. Then, several different liquids with known and varying surface tensions are applied as drops to a sample. The penetration time of each is recorded and plotted against its surface tension. A line is fitted through the points for the sample from which the surface tension associated with a 25 second penetration time is determined. This is denoted as the critical surface tension. The values for each sample are then able to be compared. The lower the critical surface tension, the higher the initial repellency of the soil.

Any of the three preceeding techniques may be used to characterize the relative extent of water repellency in a series of soil samples. The sequence used in this investigation is as follows. The Water Drop Penetration Time is used as a quick, initial test to determine the success or failure of a sample. Then, the critical surface tension method is used to rank the samples and distinguish the two most hydrophobic. Finally, the liquid-solid contact angle is measured for those two samples for comparison against the original.

Development of Theories

The common factor in the early observations was the presence of an organic material believed to coat the soil in some manner. The theory was tested by treating clean sand with ammonium hydroxide (NH_4OH) extract of the chaparral litter (Letey et al., 1962b). A noticable increase in the contact angle resulted, indicating that the organic component did indeed cause some degree of water repellency in the sand. The subsequent investigations with regard to soils subjected to fire (Krammes and DeBano, 1965; DeBano and Krammes, 1966) further clarified the issue. It was confirmed that hydrophobic behavior is not a result of low moisture content due to heating. Also, it is not related to grain size or pore opening dimension. In fact, there was a general tendency for repellency to decrease as the size class decreased. These discoveries, among other things, led to the conclusion that wetting resistance is actually a result of organic coating on the mineral soil particles which alter the liquid-solid contact angle.

These studies also examined the specific effects of heating upon hydrophobic properties. Even though the organic materials themselves do result in a certain extent of repellency, it was found that heating such soils over various temperatures and durations greatly enhanced

the condition. Then, after reaching a maximum, the repellency quickly declined and eventually was totally eliminated following additional heating. It was presumed that the organic material was actually burned and destroyed by the excess heating. It was apparent, therefore, that an optimum range of heating exists for which ultimate hydrophobic behavior is induced in the soil.

These developments led to the following scenario (Krammes and DeBano, 1966; DeBano, 1969b). Some plants must contain substances that are capable of making a soil non-wettable. Logically, these substances are also found in the litter layer beneath the plants. Normal decomposition and rainfall cause them to mix with and coat the upper crust of the soil (see Figure 5A). This results in a mild degree of water repellency located at the surface, but it is not impenetrable. After being subjected to the intense temperatures of a wildfire, the litter layer is destroyed and a water repellent zone is formed a few inches below the surface (see Figure 5B). This zone has been found to be much more resistant that the original surface layer.

Two mechanisms were assumed to contribute to this phenomenon. First, the fire temperatures are high enough to destroy the water repellency in the surface region. The depth to which this occurs depends on the temperature

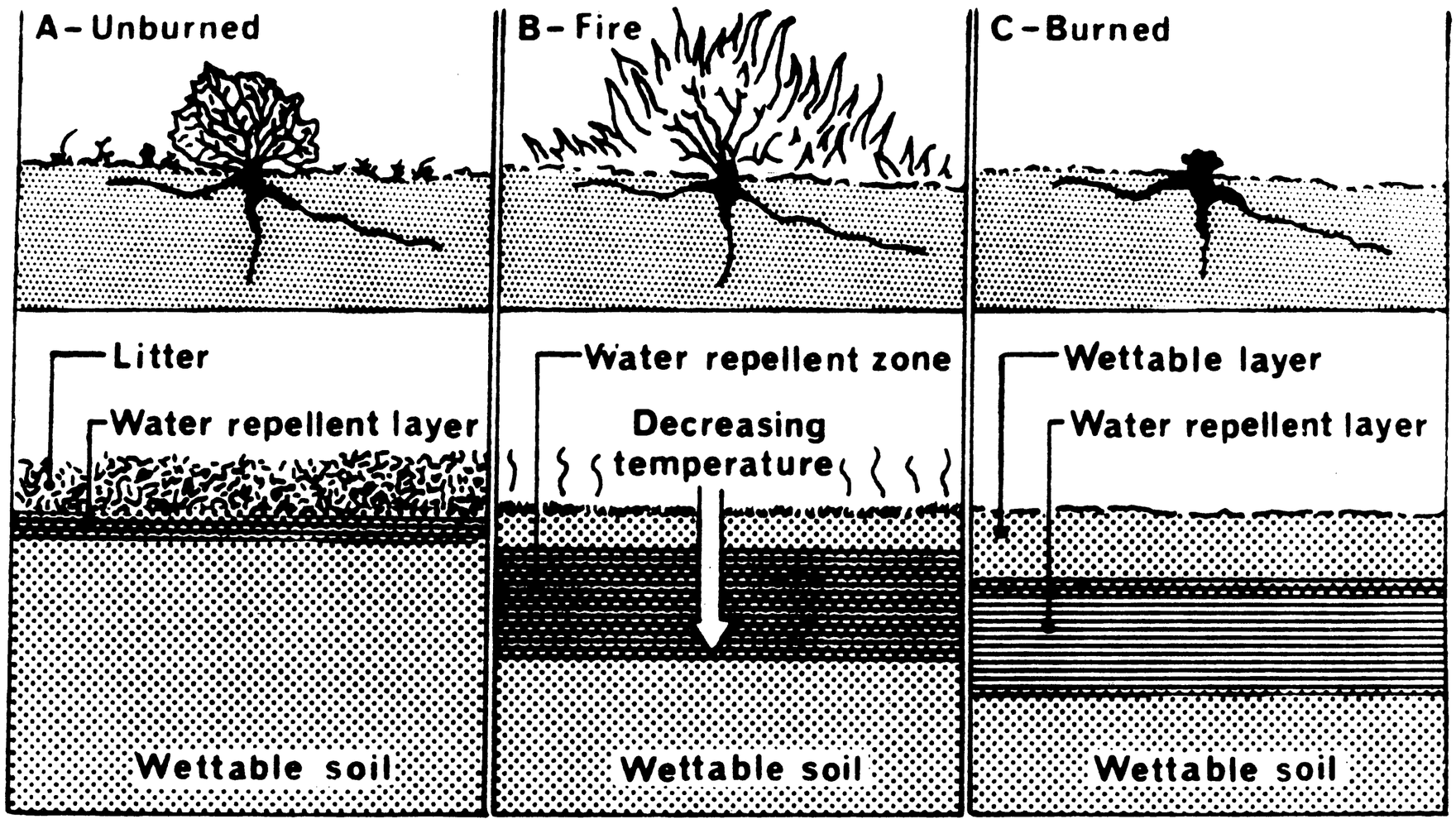


Figure 5. Water repellency before, during, and after fire. (A) Before fire, the hydrophobic substances accumulate in the litter layer and mineral soil immediately beneath it; (B) fire burns vegitation litter layer, causing the hydrophobic substances to move downward along temperature gradients; (C) after a fire, a water repellent layer is located below and parallel to soil surface on the burned area. (After DeBano, 1969b)

and duration of heating. There exists, therefore, a depth beyond which the heating conditions are such that the water repellency is not destroyed, but enhanced. Second, the formation of this layer involves some sort of downward diffusion of vaporized hydrophobic substances from the surface. It is assumed that temperature gradients exist that volatize the substances and cause them to move downward where they are able to condense. In this manner, an extremely hydrophobic layer is produced several inches below the soil surface (see Figure 5C).

The primary conclusion to be extracted from this concept is that inherent non-wettability must be present before heating will have a significant effect. Subsequent investigations (Savage et al., 1969) were able to modify this theory and define more precisely the state of inherent non-wettability. It appears that hydrophobic substances do not come directly from plants. Instead, they are found in situ and are directly related to litter decomposition and fungal growth. Therefore, a range of fungal products and other organic materials must be present prior to the heating mechanism. Another clarification of the theory indicates that the organic vapors are produced by a chemical reaction such as pyrolysis (heat-induced chemical change) rather than a physical

vaporization. All of these facts lead to another important conclusion. Heat-induced water repellency is a universal property of soils and is a function of those soil factors affecting fungal proliferation and litter decomposition.

More recent work (Jex et al., 1985) has isolated some of these factors, particulary humidity and saturation. Sandy soils incubated at 100% relative humidity (RH) were observed to have a sharp increase in water repellency. The primary cause of this was discovered to be the growth of actinomycetes, a filamentous type of bacteria. However, if the soil was wetted, the penetration time immediately fell to zero. A decline in penetration time also occurred if the soil was maintained for long periods in a low humidity (less than 90% relative humidity) environment. With this in mind, another theory was developed for a naturally occurring water repellent sandy soil. Such a soil will lose its wetting resistance after being wetted. As it dries, but with the relative humidity maintained near 100%, microbial activity becomes high and the resistance dramatically increases. After it dries below 90% relative humidity, penetration time gradually decreases as a result of a nonbiological mechanism. Water repellence may only be restored if relative humidity is returned to 100%.

The most plausible explanation, however, was offered by a somewhat earlier study (Miller and Wilkinson, 1977). While investigating the causes of nonwettable spots on golf greens, the nature of the organic coating itself was determined. Various extraction techniques isolated the material which in turn was identified by obtaining an infrared spectrum. The material was concluded to be soil fulvic acid, a type of humic substance. There is a distinct relationship between fungal activity and the synthesis of humic materials (Martin and Haider, 1971). Also, it was further concluded that the nonwettability of the fulvic acid materials probably occurs because of the formation of calcium and magnesium fulvate which become hydrophobic after a prolonged drying cycle. The role of humic substances in this process will be discussed in greater detail in a subsequent section.

Chemical Considerations

Before examining humic substances, a brief, general review of the chemistry of the materials that make soils hydrophobic is in order. In an effort to create an artificial treatment process, research was conducted into the particular chemical characteristics required for hydrophobic behavior in soils (Bozer et al., 1969). Since it was known that phenolic compounds are very common in natural resins and vegetative matter, the focus of attention was placed on a class of compounds known as substituted phenols (see Figure 6). It was also known that chemical structures that waterproof soils must contain hydrophilic groups that absorb on soil grains and hydrophobic groups that extend from soil grains to form a "new surface" with altered wettability characteristics. This indicated that the type and positioning of the substituted groups dictated the ability of the final compound to perform this task.

Specifically, hydroxy- and amino- substituted phenolic compounds were investigated. Figure 6 clearly shows the importance of the location of the substituted group on the benzine ring. In both cases, the compound became active only when the group was positioned on the carbon atom adjacent to the existing hydroxyl group. These compounds are known as substituted catechols (-OH)

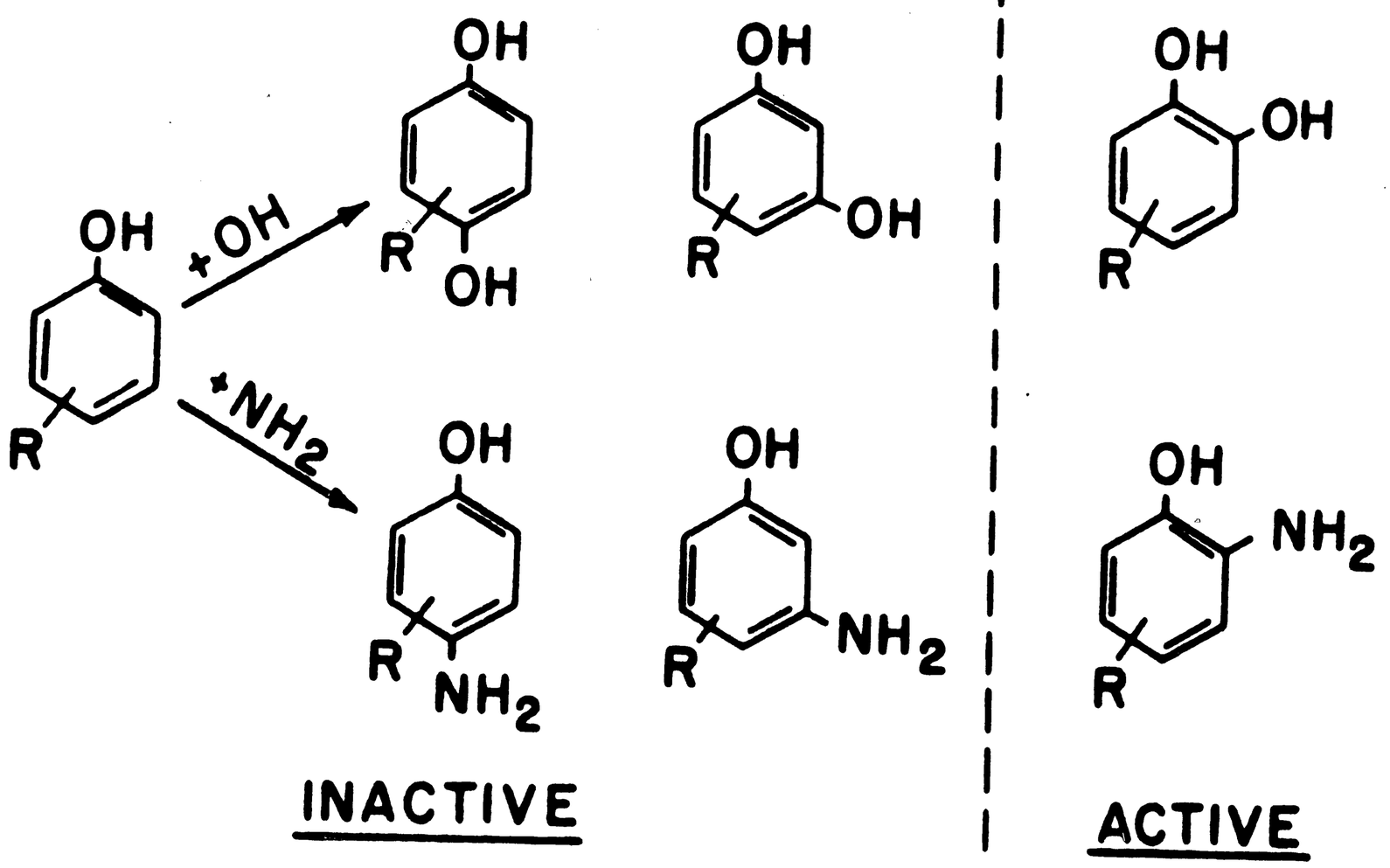


Figure 6. Soil waterproofing as related to positioning of second hydrophilic group in phenolic materials. (After Bozer et al., 1969)

and aminophenols ($-NH_2$), respectively. Once this had been established, it was further determined that the waterproofing ability of these compounds also depended upon the structural characteristics of the substituents indicated by the R in Figure 6. In general, compounds having C_3H_7 or larger substitutions, reaching a maximum at about 8 carbon atoms, were found to become increasingly active. Finally, other substituted phenols have exhibited such activity, but in all cases the substituents are located on adjacent carbon atoms within the benzine ring.

Aside from structural configuration, the other important chemical factor is the method of adsorption utilitized by the hydrophobic substance (Thurman, 1985). The first important method is hydrogen bonding (see Figure 7). These primarily consist of the bonding of nitrogen containing organic bases to silica (sandy soils) and organic acids to alumina (clays). Another method is cation exchange, which usually involves expanding clay minerals such as montmorillite. The most significant method for our purposes, however, is the complexation of metal ions (see Figure 8). Most metal ions may accept more than one pair of electrons and each pair corresponds to a donor atom. The significance that this has with regard to humic substances will be detailed in the following section.

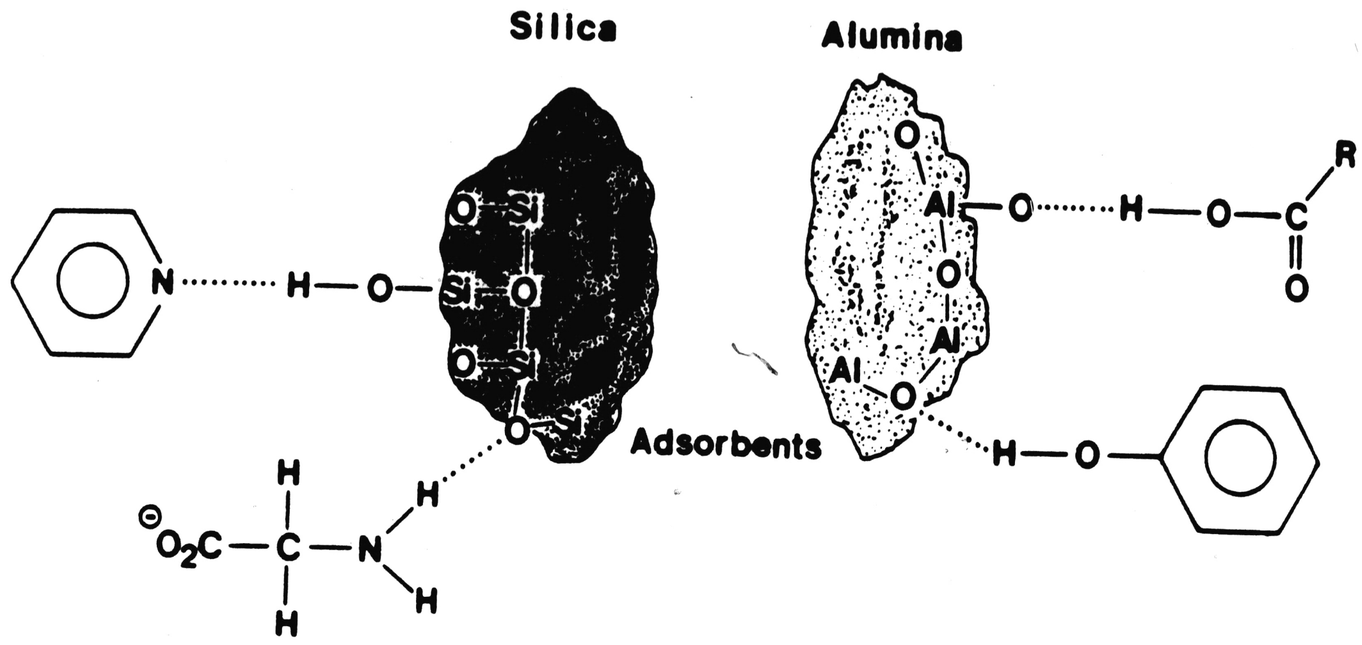


Figure 7. Two important types of hydrogen bonding involving natural organic matter and mineral surfaces in sediment. (After Thurman, 1985)

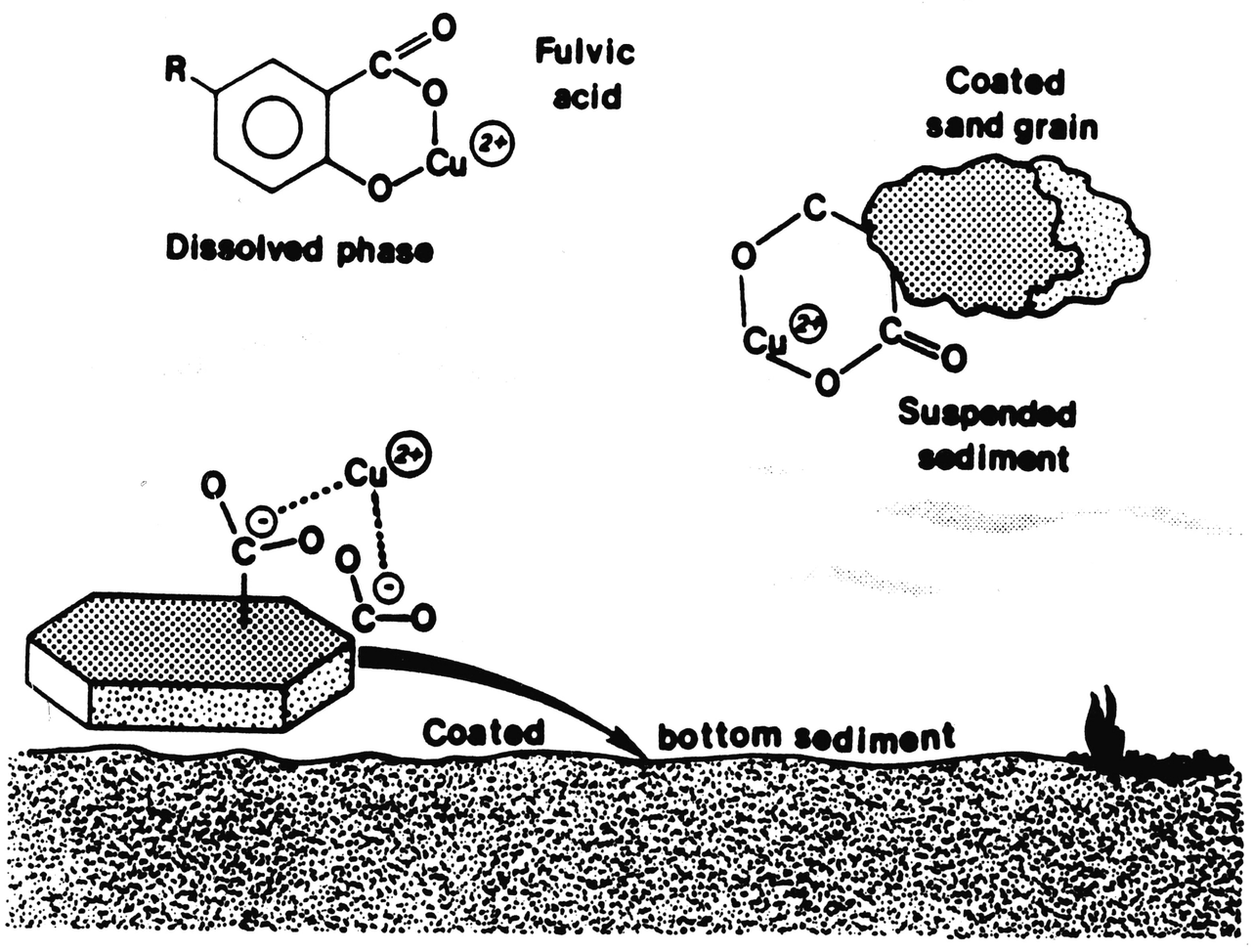


Figure 8. Complexation of metal ions by organic matter in suspended sediment, bottom sediment, colloidal, and dissolved phases. (After Thurman, 1985)

Humic Substances

Humic substances are defined as a general category of naturally occurring, biogenic, heterogeneous organic materials that can generally be characterized as being yellow to black in color, of high molecular weight, and refractory (Aiken et al., 1985). They consist of three major fractions which are defined in terms of their solubilities. The first is humin, and it is insoluable in water at any pH value. Humic acid is that fraction that is insoluable in water under acidic conditions (below pH 2) but becomes soluable at greater pH. Finally, fulvic acid is the fraction which is soluable under all pH conditions. The latter two are of primary concern and some of their classification and chemical properties are shown in Figure 9.

The formation of humic substances in soil is a multistage process (Stevenson, 1985) including:

(1) Decomposition of all plant components into simplier monomers,
(2) Metabolism of the monomers with an accompanying increase in the soil biomass,
(3) Repeated recycling of the biomass carbon with synthesis of new cells, and
(4) Concurrent polymerization of reactive monomers into high-molecular-weight polymers.

It is believed that polyphenols synthesized by micro-organisms are polymerized alone or in the presence of amino compounds to form brown colored polymers. This

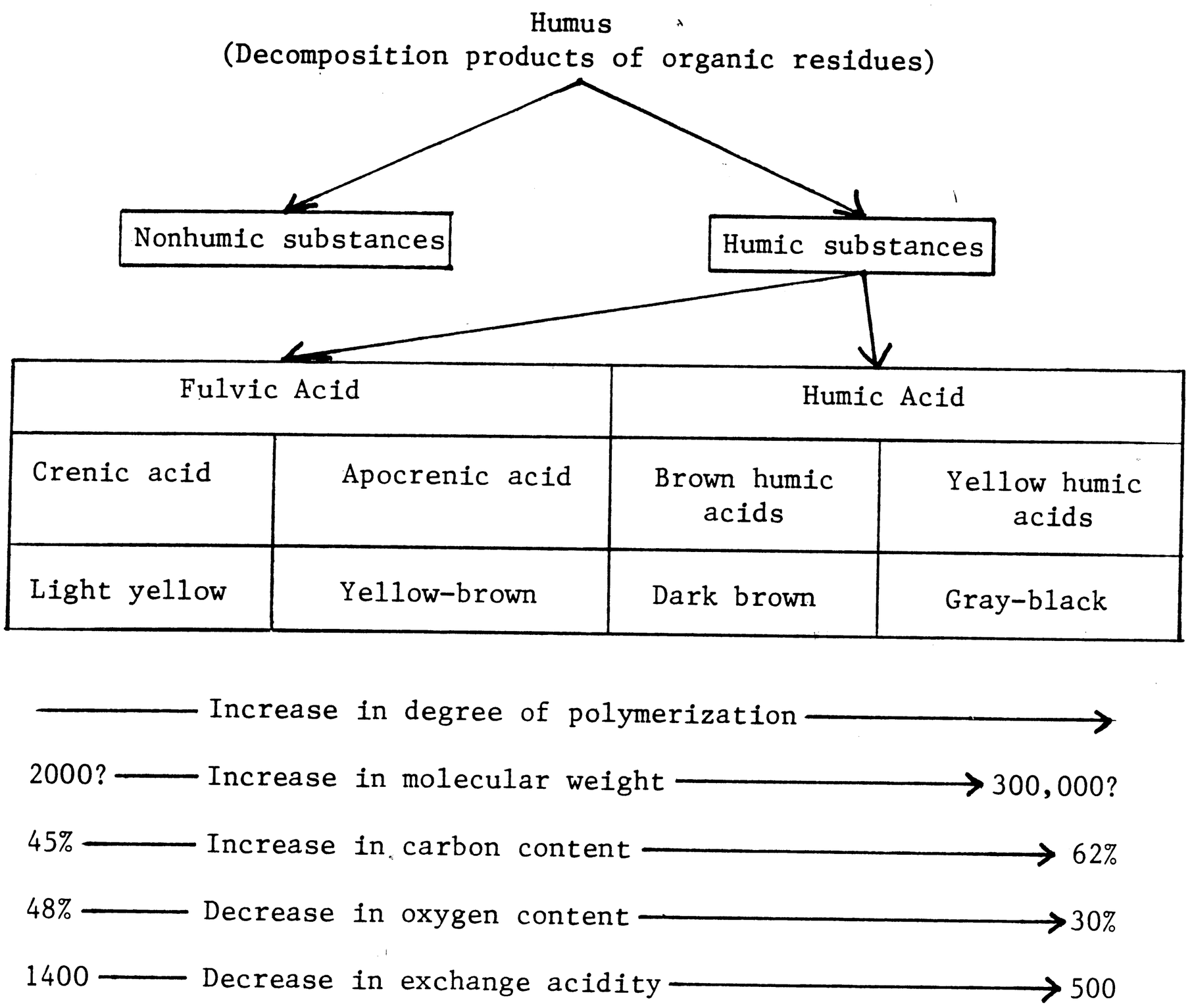


Figure 9. Classification and chemical properties of humic substances. (After Stevenson, 1985)

hypothesis is consistant with the previous concepts regarding the creation of an organic material in the presence of fungi within the upper soil layer. It is likely, therefore, that this material was a humic substance.

The structural makeup of the humic substances are similar to the general description given previously. Substituted phenols are in abundance with the functional groups primarily consisting of oxygen- containing acidic groups such as COOH (carboxyls) and OH. Also, these groups are in the proper location with respect to the free OH group - on adjacent carbon atoms within the benzine rings (Stevenson, 1985). For humic acid, the basic structure of the "type" molecule consists of an aromatic ring of the di- or trihydroxy- phenol type bridged by - O -, - CH_2 -, - NH -, - N -, or - S - groups and containing both free OH groups and the double linkage of quinones (see Figure 10). The type structure for fulvic acid consists of aromatic and aliphatic components extensively substituted with oxygen-containing functional groups (see Figure 11). Note the particular abundance of COOH (carboxyl) groups.

Humic and fulvic acids have a profound ability to form stable complexes with metal ions. This can be attributed to their high content of oxygen- containing

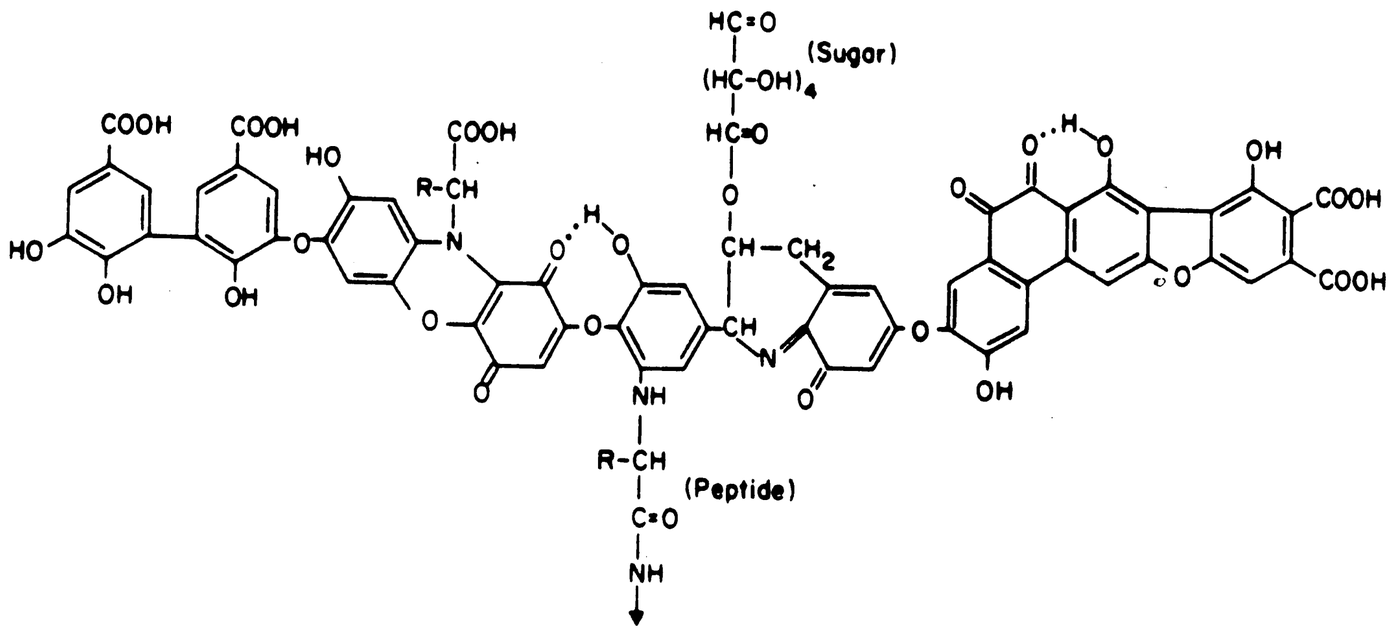


Figure 10. Hypothetical (type) structure of humic acid showing free and bound phenolic OH groups, quinone structures, oxygen as bridge units, and carboxyls variously placed on the aromatic ring. (After Stevenson, 1985)

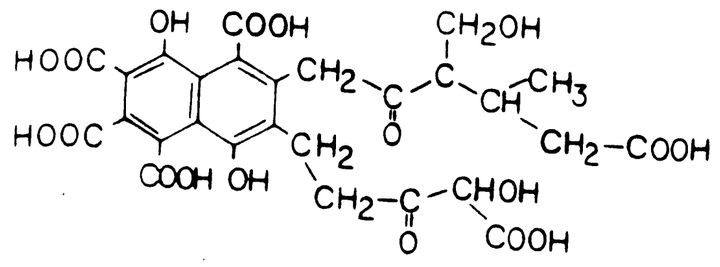


Figure 11. Type structure of fulvic acid. (After Stevenson, 1985)

functional groups, particularly COOH (Stevenson, 1985). In this way, they can be adsorbed onto mineral soils as macromolecular complexes bound together by di- and trivalent cations, or by hydrogen bonding. The main polyvalent cations responsible for this are Ca^{2+}, Fe^{3+}, and Al^{3+}. This type of bonding is usually associated with clay minerals and such an aggregation is usually called a clay-metal-humus complex (see Figure 12). However, similar bonds are possible with sandy soils, particuarly involving the Si^{2+} cation (Thurman, 1985). Finally, these complexes with polyvalent cations tend to make the humic substances less soluable than before, particularly humic acid (Stevenson, 1985). Factors affecting the degree of solubility include the extent to which the complex is saturated with metal ions and the pH of the surrounding fluid.

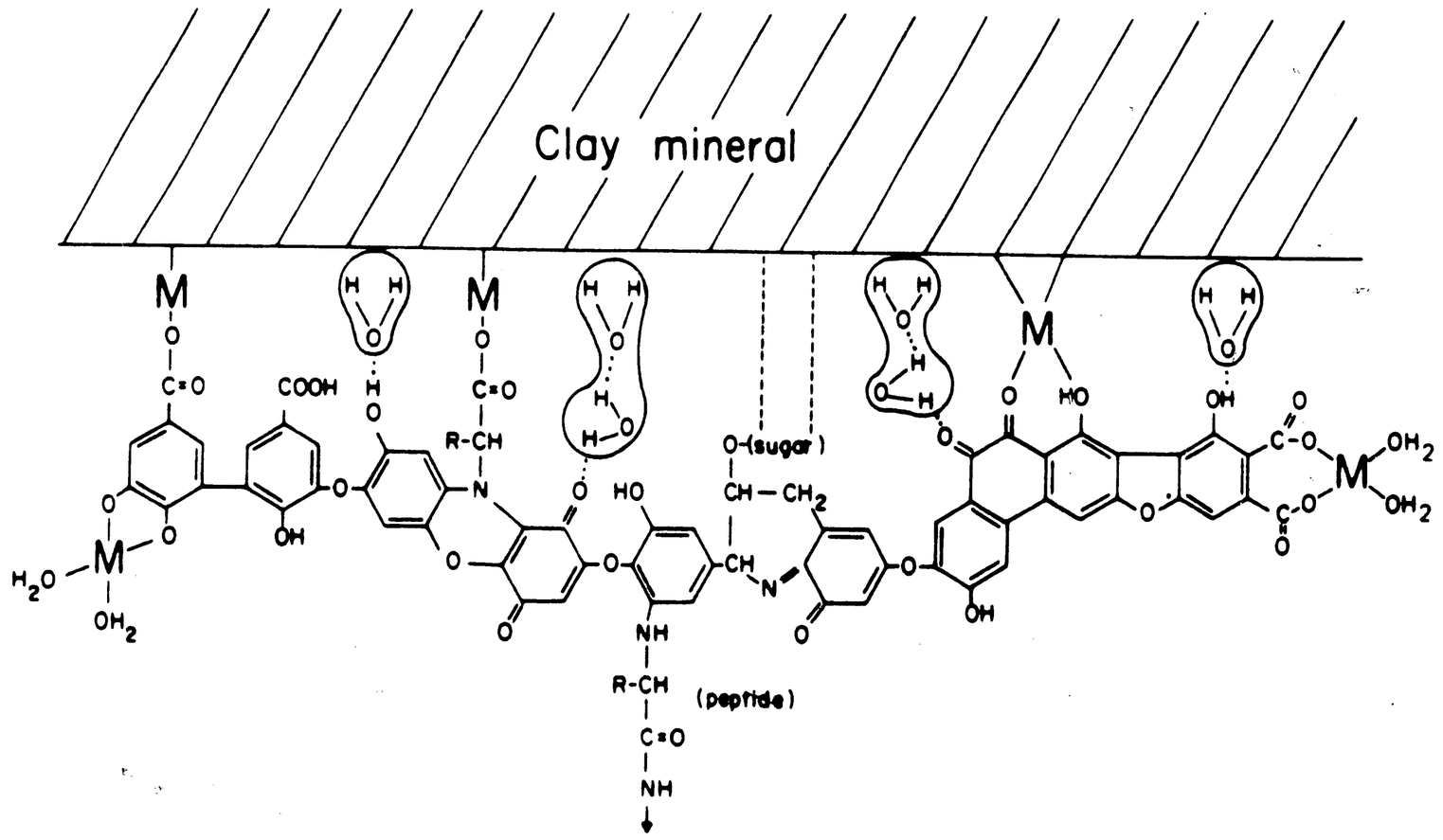


Figure 12. Schematic diagram of a clay - humate complex in soil. (After Stevenson, 1985)

Activated Sludge

Considering all of the aforementioned evidence, it is apparent that some type of humic substance is the best choice for artificially reproducing the hydrophobic effect in soils. A likely procedure would involve the mixing of the humic substance in an aqueous solution with a certain quantity of soil. In this way, a uniform distribution of these molecules would permit a relatively even rate of adsorption throughout the soil matrix. Theoretically, then, the surface characteristics of the soil particles would be altered, resulting in a significant change in the liquid-solid contact angle. Such an occurrence would render the soil hydrophobic.

However, the primary purpose of this investigation is to develop a low-cost alternative to existing low-permeability soils. Unfortunately, pure solutions of humic or fulvic acid are fairly difficult to extract and are not in great demand. This combination currently prevents them from being a cost effective solution. Although an extensive application such as artificially produced hydrophobic soil may change that, it was decided to pursue a more guaranteed low cost material at this time. In this material, two major criteria had to be met. First, it had to be similar chemically to humic substances. Second, it had to exist in great abundance,

preferably as a waste product.

The logical conclusion was activated sludge. In the activated sludge process, a suspended aerobic microbial culture is used to treat waste water (Tchobanoglous and Schroeder, 1985; Viessman and Hammer, 1985). These microorganisms (mostly bacteria) metabolize and biologically flocculate the organics. They then, along with their waste products, settle from the aerated mixture (see Figure 13). This settled material is what is known as activated sludge. It is the waste products of the microorganisms that contain the organic compounds similar in chemical structure to humic substances. In fact, it is possible that humic substances themselves exist within activated sludge. Finally, it is quite obvious that activated sludge exists in great quantities and is a waste product which requires disposal in an effective manner. It is with these facts in mind that activated sludge is chosen for use in this investigation.

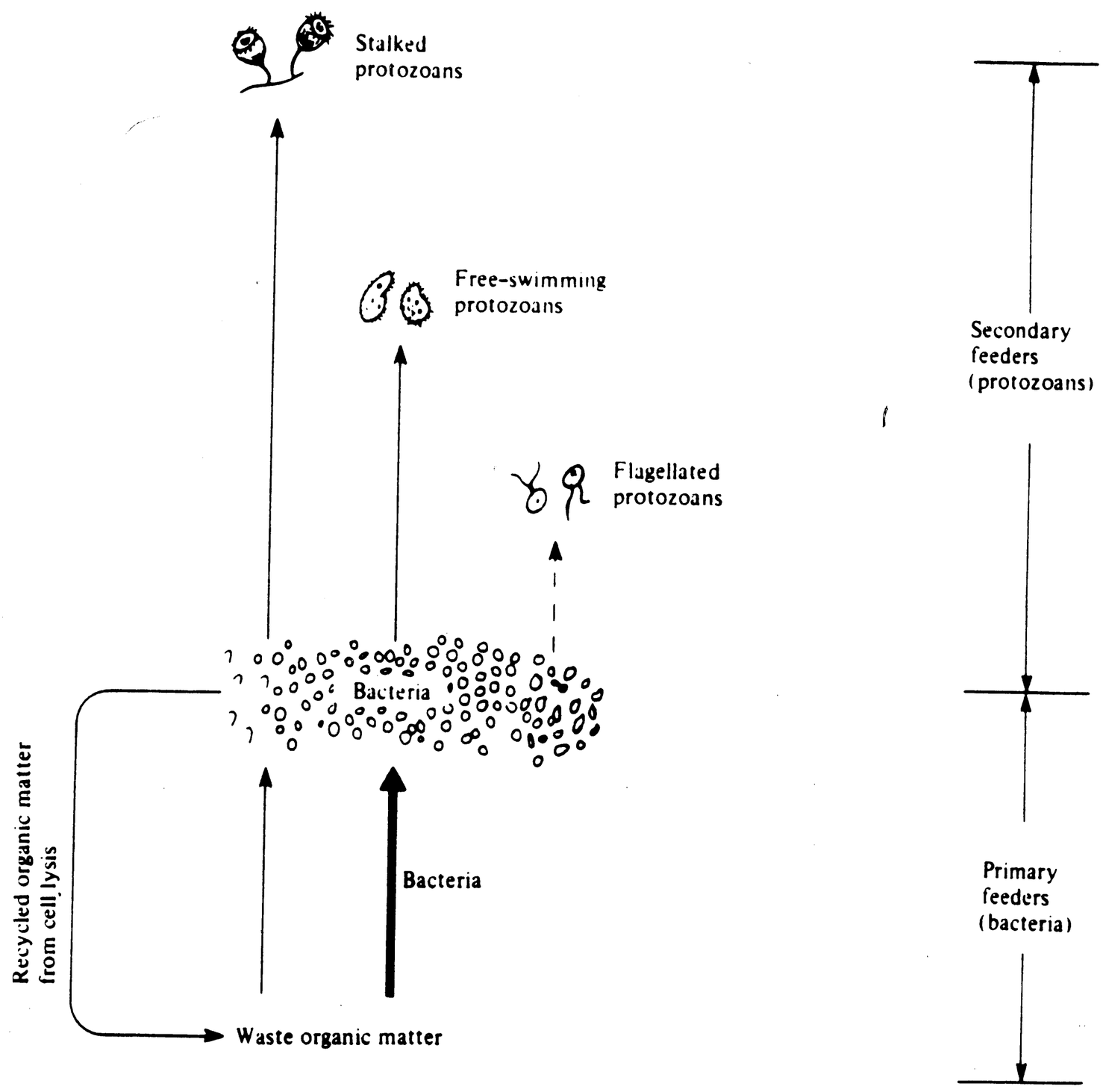


Figure 13. Schematic diagram of the population dynamics in activated sludge indicating the role of bacteria. (After Viessman and Hammer, 1985)

III. EXPERIMENTAL PROCEDURE

In keeping with the low-cost aspect of this project, it was important to use a soil that would not require any significant pre-treatment preparation methods. In fact, the intention of this proposed procedure is to perform it on soils found at the construction site where the product will be used. This will eliminate transportation and excessive storage expenses. With this in mind, a random sandy soil was chosen from a local (Bethlehem, PA) construction site. A large sample was removed and allowed to air dry. Standard soil classification tests were then conducted, including mechanical and hydrometer grain size analysis and Atterberg Limit determinations (ASTM D423, D424). Since the hydrophobic effect becomes more pronounced in sandy soils, it was important to verify this condition.

The activated sludge was obtained from the Allentown, Pennsylvania Waste Water Treatment Plant at Kline's Island. A quantity (about 10 gallons) of raw sludge was removed at random from a pumping facility within the plant. It was black in color, fairly thick in consistency, and appeared to have several solid particles in suspension. The sample was easily obtainable and qualified as an inexpensive source for humic substances.

A portion of the sandy soil was spread in thin, flat layers in several shallow mixing pans. The weight of soil used was carefully measured and kept constant to control the mixture proportions. Similarly, specific amounts of the well-agitated sludge were added to the soil. First, an adequate amount was placed in the first pan to completely cover the soil and then equal amounts were placed in the others. Working near proper ventilation apparatus, the soil and sludge were thoroughly mixed with a spatula. The mixtures were then reweighed and placed aside to air dry. After 3 days, they were weighed again. Initial moisture content and percent solids added were computed from this data.

The treated soil was separated into several individual containers for testing. Since it was known that water repellency in organically-coated soil is dependant upon heating temperature and duration, three of each were chosen. Temperatures of 100°C, 200°C, and 300°C and durations of 5, 10, and 15 minutes were selected to coincide with similar earlier investigations (Savage et al., 1969). Also, previous work by the author (Gauffreau et al., 1987) had indicated that higher temperatures or longer durations were not justified. This combination, therefore, required that a total of eleven samples be created: one for each temperature and duration pairing,

one for the treated but unheated soil, and one for the original, untreated soil.

Heating was accomplished with a Thermolyne® variable temperature furnace. Then, at intervals of 5 minutes, one was removed and placed aside to cool. In this way, the nine samples were prepared. After cooling, these nine were tested along with the other two samples by the simple water drop test. A "standard size" drop of distilled water from a medicine dropper was carefully placed on the soil. The time taken by the drop to completely penetrate the sample was measured by the stopwatch and recorded to the nearest second. This time is referred to as the Water Drop Penetration Time (WDPT), which was used to determine the relative success or failure of the treatment technique.

The critical surface tension approach was used to classify each sample in order from most to least hydrophobic (Watson and Letey, 1970). The critical surface tension was defined as the surface tension of a fluid which will cause a drop of that fluid to remain on the soil for a specified duration. A series of aqueous solutions were prepared with known and varying surface tensions. Since the Water Drop Penetration Times for water range from 0 (zero) seconds to several minutes, it was apparent that solutions with surface tensions both

SURFACE TENSIONS

(τ = surface tension in dynes/cm)

Surface Tension of Ethyl Alcohol
in Water (@ 40° C)

(% = volume % of alcohol)

%	5.00	10.00	24.00	34.00	48.00	60.00	72.00	80.00	96.00
τ	54.92	48.25	35.50	31.58	28.93	26.18	24.91	23.43	21.38

Surface Tension of Sodium Hydroxide (NaOH)
in Water (@18° C)

(% = weight % of NaOH)

%	2.72	5.66	16.66	30.56	35.90
τ	74.35	75.85	83.05	96.05	101.05

Table 1. Surface tensions of various aquious solutions.

greater and less than that of water would be required to obtain that specified duration. Water has a surface tension of 72.1 dynes/cm, so various concentrations of sodium hydroxide (NaOH) and ethyl alcohol were used to obtain larger and smaller surface tensions, respectively (see Table 1). Drops of each solution were placed on a sample until an adequate number of penetration times surrounding the specified duration were obtained. A simple plot of penetration time versus surface tension reveals the critical surface tension for that sample. In this manner, all eleven samples could be ranked by recalling that the lower the critical surface tension, the higher the initial repellency.

The two most hydrophobic samples as determined by the above procedure were then isolated for further testing against the original, untreated and treated, unheated samples. As discussed earlier, the physical property that best defines the extent of water repellency in soil is the liquid-solid contact angle. In order to measure this, it is assumed that soils can be represented as cylindrical capillaries (Letey et al., 1962; Letey, 1969). Flow may be approximated by:

$$Q = \frac{\pi r^4 P}{8 L \eta} \qquad (2)$$

where Q is the rate of flow in volume per unit time;

P, the pressure driving the water;

r, the radius of capillary;

η, the viscosity of the solution;

L, the capillary length.

The pressure (P) may be evalutated in terms of its capillary and gravitational components. The pressure due to capillary force is:

$$P_c = \frac{2\ T \cos \theta}{r} \qquad (3)$$

where θ is the liquid-solid contact angle and

T is the surface tension of the fluid.

The pressure due to gravity is:

$$P_g = \rho\ g\ h \qquad (4)$$

where ρ is the density of the fluid;

g, the gravitational constant; and

h, the capillary height above the level (head) of the fluid reservoir.

Figure 14 shows the configuration of the apparatus used for sandy soils. At equilibrium of capillary rise, the pressures due to capillary force (P_c) and gravity (P_g) are equivalent. Setting equation (3) and equation (4) equal to each other and solving for h, the height of capillary rise is given by:

$$h = \frac{2\ T \cos \theta}{\rho\ g\ r} \qquad (5)$$

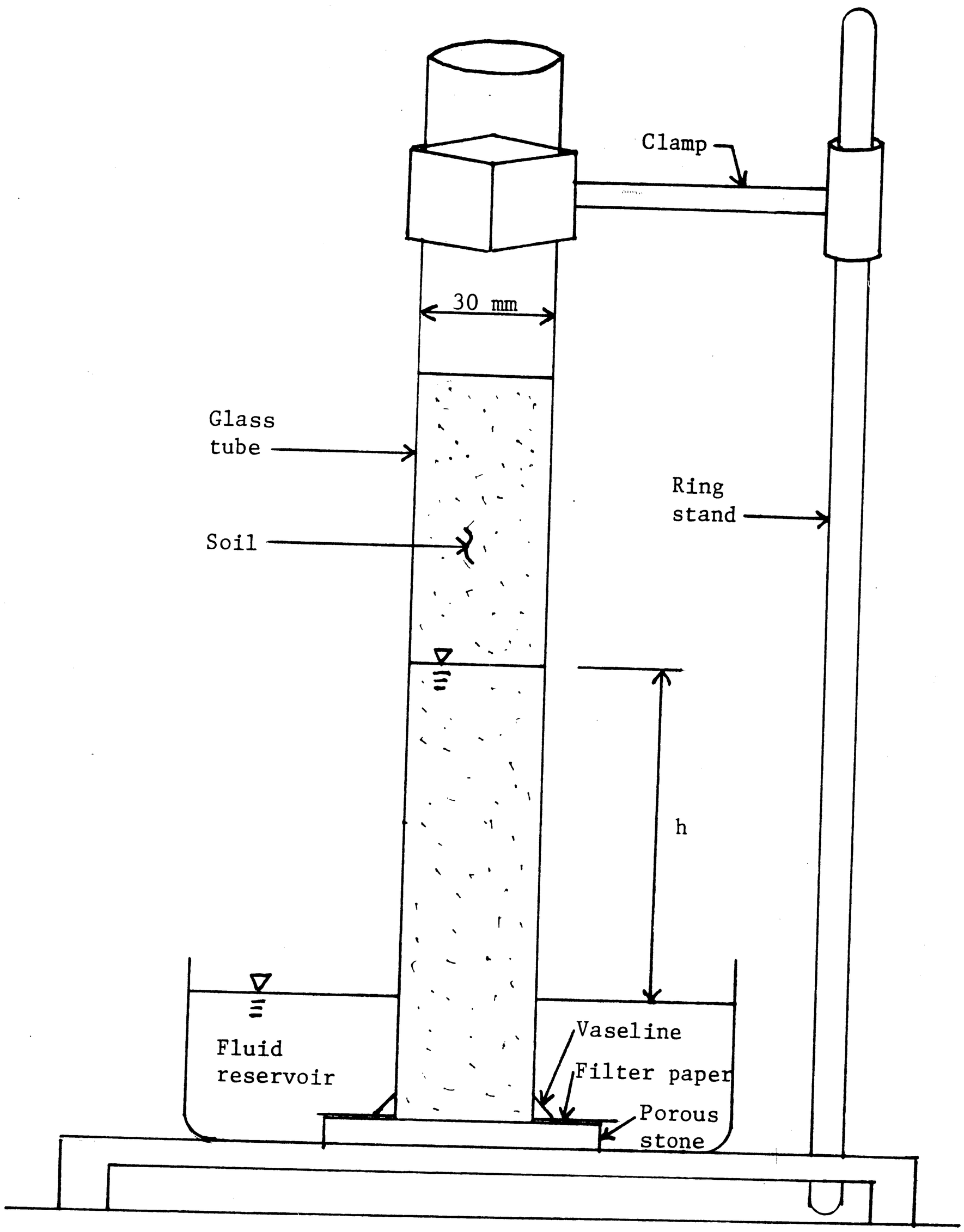


Figure 14. Schematic diagram of capillary rise device used for liquid-solid contact angle measurement.

Only two factors (r and θ) in equation (5) are not known nor readily measured. Since it is assumed that the liquid-solid contact angle between ethanol and a solid is zero (Letey et al., 1962a), ethanol may be used as the fluid to determine r. In other words, if $\theta = 0$ and h is measured for ethanol, r may be computed from:

$$r = \frac{2\ T_E}{\rho_E\ g\ h_E} \qquad (6)$$

This calculated r value may then be used to find 0 for other pore fluids, particularly water. In this case, the liquid-solid contact angle (θ) is given by:

$$\cos\ \theta = \frac{h_w \rho_w g\ r}{2\ T_w} \qquad (7)$$

The test procedure consists of packing the soil sample into a glass tube with a porous stone and filter paper at the bottom to retain the particles (see Figure 14). The tube was 30 mm in diameter and 50 cm in length. The inner surface of the tube was treated with paraffin to prevent the tube from being wet more than the soil. Packing was achieved by pouring the soil in equal lifts and dropping the tube an arbitrary (but constant) number of times. The tube was then stood with its bottom in a pan of fluid (ethanol and then distilled water for each sample). The area of the pan maintained the surface of

the fluid reservoir at a relatively constant level. The height of capillary rise was checked periodically and equilibrium was found to be reached after 24 hours. The final heights were then used in equation (6) and equation (7) to determine the liquid-solid contact angle.

The results of the liquid-solid contact angle test were used to isolate the most hydrophobic sample. This sample was then examined for permeability characteristics and compared to those of the original sandy soil. A significant reduction in the permeability is the most relevant indication if this new hydrophobic soil is to be considered as an alternative liner material. First, the coefficient of permeability, k, was established for the original, untreated sandy soil using a conventional method. The constant head test was used to estimate k for the untreated granular soil (Bowles, 1978). The line details of the test are shown in Figure 15. The coefficient of permeability is computed as:

$$k = \frac{Q\ L}{A\ h\ t} \quad cm/s \qquad (8)$$

Since k is a function of the viscosity of the pore fluid (water, in this case), and viscosity is a function of temperature, a temperature correction must be applied to the apparent k computed from equation (8). The coef-ficient of permeability is standardized at 20 C, so the

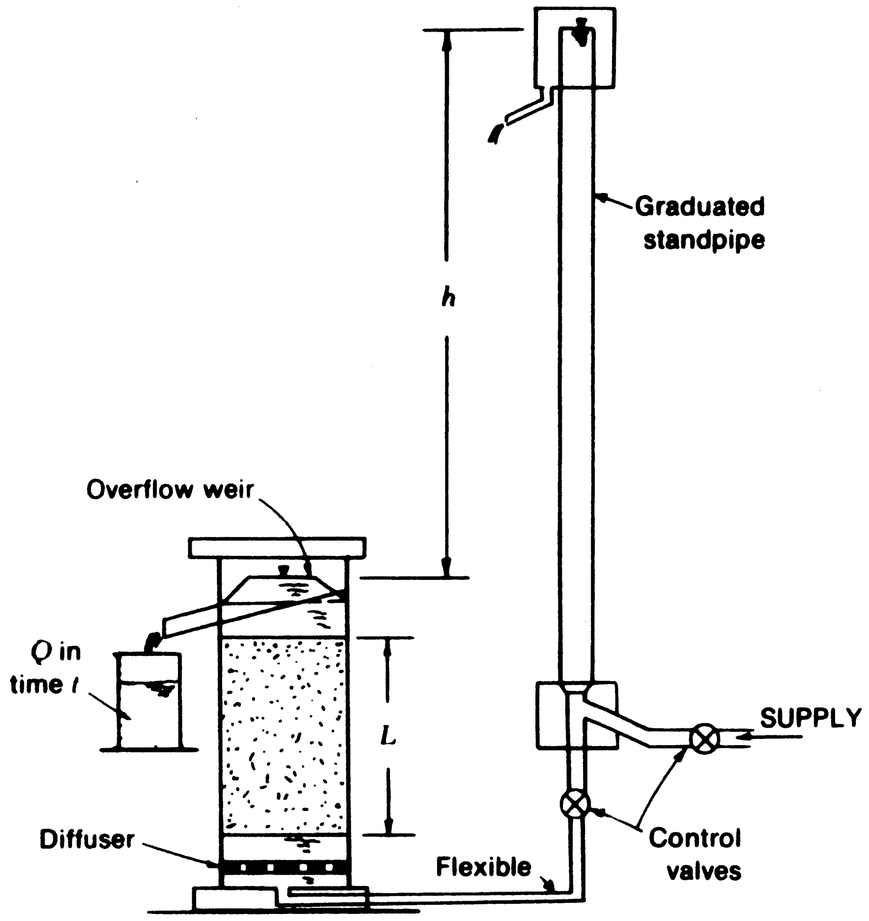


Figure 15. Line details of the constant head test. (After Bowles, 1978)

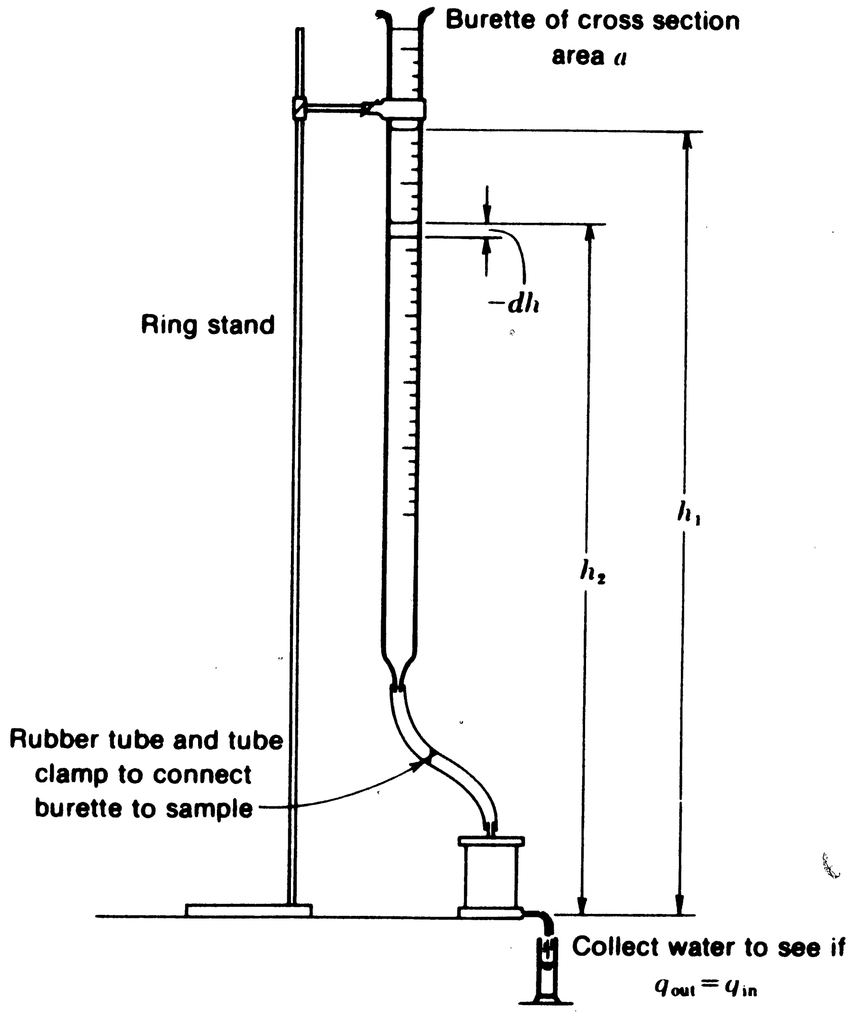


Figure 16. Line details of the falling head test. (After Bowles, 1978)

appropriate correction factor is taken from Table F-1 in Appendix F. Finally, k also depends the void ratio, e, of the soil. Since a change in k of several orders of magnitude was possible, a similar compaction method was used to simulate that used in the following test for compatibility purposes.

The simple constant head test was not used for the hydrophobic soil. Several anticipated factors required the use of a modified long-term triaxial permeameter cell developed at Lehigh University (Evans and Fang, 1985). Those factors included the relatively long time required for saturation and a change in k over time. The Lehigh triaxial permeameter cell (see Figure 17) has several unique features that permit this and other types of testing. Primarily, the permeant may be changed and the inflow and outflow riser tubes may be filled or emptied without changing the state of stress on the sample. Also, it has the ability to measure both inflow and outflow volumes for permeants with pH values varying from 1.0 to 12.6 without damage to the apparatus because the parts in contact with the permeant are made of teflon®. Finally, the calibrated riser tubes provide an effective method for measuring the change in permeability over time.

The most hydrophobic sample was reproduced in a

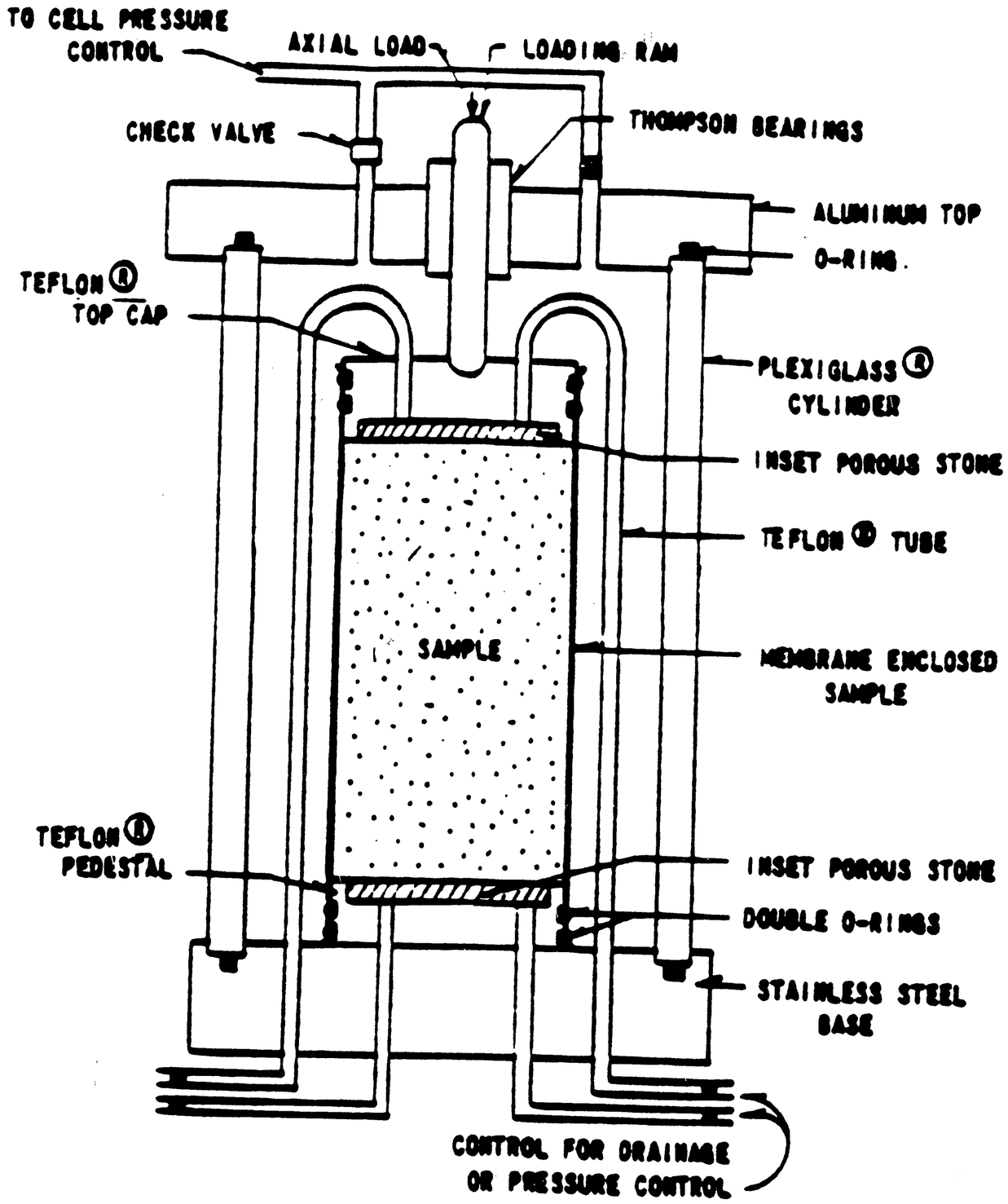


Figure 17. The Lehigh Long-Term Triaxial Cell Permeameter for permeability testing with hazardous and toxic permeants. (After Evans and Fang, 1985)

large enough quantity to fill a mold for the Lehigh permeameter. It was placed dry in the membrane and compaction was achieved by tamping and maintained by applying a vacuum pressure. After placing the specimen in the apparatus and beginning the test, the time taken for complete saturation was noted. Next, the initial coefficient of permeability was measured and calculated by simulating a simple falling head apparatus (see Figure 16 for a line diagram). Here, the coefficient of permeability is computed as:

$$k = \frac{a\ L}{A\ t} \ln \frac{h_1}{h_2} \quad \text{cm/sec} \qquad (9)$$

The test was permitted to run for several days. It was an assumed possibility that the organic coating could dissolve over time. This would revert the liquid-solid contact angle to its original value and thereby cause the permeability to increase.

Finally, Scanning Electron Microscope (SEM) photographs were taken of the original, untreated sandy soil and the most hydrophobic soil as used in the permeameter. It was anticipated that these pictures would show the organic coating and any physical characteristics that it may impart to the soil particles.

IV. RESULTS and DISCUSSION

Classification

The randomly selected, local soil was initially tested for classification purposes. It was visually observed to be a light brown sandy clay. After passing the soil through a number 4 U.S. standard sieve (4.75 mm diameter), the Atterberg Limit Tests (ASTM D423, D424) were conducted with the following results:

Liquid limit = 33.5
Plastic limit = 22.0
Plasticity index = 11.5 (See Appendix A)

Standard mechanical sieve and hydrometer tests (ASTM D421-58 and D422-63) produced the grain size distribution shown in Appendix A. These results were used in the Unified Soil Classification System (USC) (ASTM D2487) to classify the soil as SC (clayey sand). According to the American Association of State Highway and Transportation Officials classification system (AASHTO M-145), the soil is classified as a A-2-6 (silty or clayey sand). Since a sandy soil was originally desired, it was apparent that the natural soil had too high a clay content to adequately test the proposed method. It was decided, therefore, to wash the soil through a number 140 U.S. standard sieve (0.106 mm diameter) to remove the silt and clay particles. This process yielded a new, light brown sandy

soil which was subjected to further grain size analysis. In this case, only the mechanical analysis was conducted which resulted in the grain size distribution shown in Appendix A. Here, the USC classification of the new soil was SP (poorly graded sand, little or no fines) and the American Association of State Highway and Transportation Officials classification was A-1-b (stone fragments, gravel, and sand). Clearly this soil was more appropriate for further testing.

Soil-Sludge Mixture

The data regarding the mixture proportions of this sandy soil and the activated sludge are shown in Appendix B. It was difficult to visually assess the effectiveness of the mixing process, so the completeness of the organic coating on all of the soil particles is not known. The water content of the soil-sludge mixture was determined to be 56.4% and the percent solids added was 2.15%. This indicated that the sludge contained very little solid by weight. It is assumed that the humic substances exist in suspension and weigh very little. The organic coatings, then, would have an insignificant effect on the overall weight of the soil.

Water Drop Test

Upon separation into the eleven samples, the soil was prepared for the Water Drop Test. For future reference, each sample was assigned a code name. The original, untreated sandy soil is simply called "original". The treated (with sludge) but unheated soil is called "treated". Each of the remaining samples is designated by its heating temperature and duration. For example, the sample heated at 100°C for 5 minutes is designated as "100/5". These labels are used throughout the remainder of this study.

The first observation to be made was the discoloring of each sample at different stages. Compared to the light brown of the original sample, the treated sample was considerably darker, indicating that some sort of coating was indeed present. The 100°C samples showed little change from this state. The 200°C samples also showed little change, but a trend was noticable toward a darker color for the longer durations. The 300/5 sample, however, was much darker than the treated sample indicating that a more significant reaction had taken place. Finally, the 300/10 and 300/15 samples were extremely dark, almost black. It appeared that the organic coating had burned at this heating range and left behind a charred residue.

The Water Drop Penetration Time (WDPT) was measured for each sample using a drop of distilled water (see Appendix C). This test yielded a quick, approximate estimation of the relative hydrophobic behavior of the soil. Figure 18 shows the dramatic differences in Water Drop Penetration Time for some of the samples. As anticipated, the original sample had an instantaneous (0 seconds) Water Drop Penetration Time, indicating a great affinity to water. The treated sample, however, had a Water Drop Penetration Time of 90 seconds. This is regarded as a significant difference. The water drop remained stationary on top of the loose soil and it was possible to see the drop "bridge" particularly large pore openings. After about 90 seconds, however, the drop began to spread slowly over a wider area and then disappear into the soil. This showed that the surface tension values were changing with time.

Heating at 100^{o}C did little to change the Water Drop Penetration Time. It actually appeared to diminish with longer durations, but the times were close enough to assume no appreciable effect. The Water Drop Penetration Time for the 200/5 sample, however, increased by a factor of six to 540 seconds. This is a significant observation. Apparently the heating process had a great effect on the organic coating for this temperature and duration.

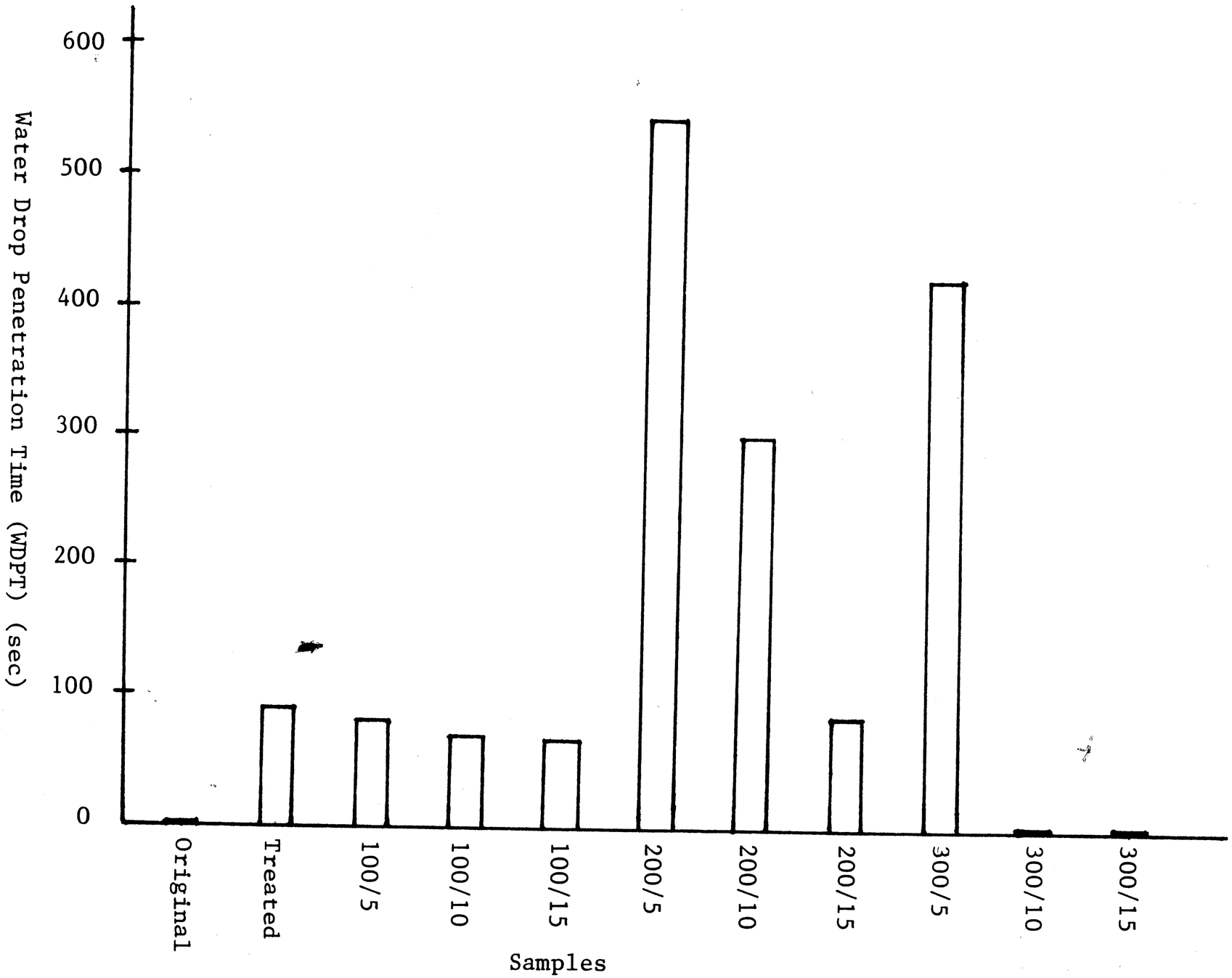


Figure 18. Bar graph of Water Drop Penetration Time per sample.

Longer heating reduced the Water Drop Penetration Times back towards 90 seconds, thereby nullifying the earlier effects. The 300/5 sample, though, had a high Water Drop Penetration Time of 420 seconds. This trend indicates that heating duration has a more significant effect than heating temperature. Finally, the burned 300/10 and 300/15 samples had instantaneous Water Drop Penetration Times. Obviously the burning at such high heating ranges destroys the organic coating completely.

Critical Surface Tension Test

The Critical Surface Tension Test was then administered to each sample. The penetration time associated with a drop of distilled, deionized water (with a known surface tension value of 72.1 dynes/cm) was found first for each (see Appendix D). After observing the distribution of these times, it was decided to use 25 seconds to define the critical surface tension. Then, depending on the initial penetration time of the water drop, drops with successively higher (NaOH) or lower (ethyl alcohol) surface tension values were tested until a range of times were found to adequately bracket 25 seconds.

The first observation is to be made with regard to the Water Drop Penetration Times. When comparing them to

those of the previous Water Drop Test, it is obvious that the new values are somewhat lower. Two factors may have contributed to this. First, distilled water was used in the original test while distilled, deionized water was used in this test. Seoncd, the Critical Surface Tension Test was performed about a month after the Water Drop Penetration Test on the same samples. It is not yet known if aging affects the organic coatings, however, in this case identical trends were observed in both tests, so the results are considered valid.

The next observation deals with the three samples (original, 300/10, 300/15) with instantaneous Water Drop Penetration Times. It was decided to attempt the solution with the maximum available surface tension value (101.05 dynes/cm), 35.9% NaOH. The penetration times for these barely reached one-half second, so it was apparent that their critical surface tensions were beyond a reasonable limit and therefore unnecessary to measure. The remaining eight samples, however, had Water Drop Penetration Times greater than 25 seconds, so solutions of ethyl alcohol were required. In all cases, the penetration times diminished to about one second for a solution of 24% alcohol (35.50 dynes/cm). This is an interesting observation as it indicates that highly alcoholic solutions are unaffected by initial water

repellency.

Plots of penetration time versus surface tension for each of the eight samples are given in Figure 19. These plots clearly show the linear nature of the reduction of penetration time with the reduction of surface tension. The difference from one sample to another is found in the slope of this relationship. The steeper the line, the smaller the critical surface angle and the higher the initial water repellency. The graphic determination of the critical surface tension (T_c) is indicated in each plot. The values range from a high of about 50 dynes/cm for the original and 100 ^{o}C samples to lows of 40 dynes/cm and 38 dynes/cm for the 300/5 and 200/5 samples, respectively. This analysis has determined, therefore, that the 200/5 and 300/5 samples are the best candidates for further testing.

Liquid-Solid Contact Angle Test

Four samples were prepared for the liquid-solid contact angle test: original, treated, 200/5, and 300/5. All four were tested simultaneously in the capillary rise apparatus shown in Figure 14. First, ethanol was used as the pore fluid. Since ethanol is assumed to wet all solids with a contact angle of 0^{o}, the capillary rise in each tube was expected to be about equal. The data in

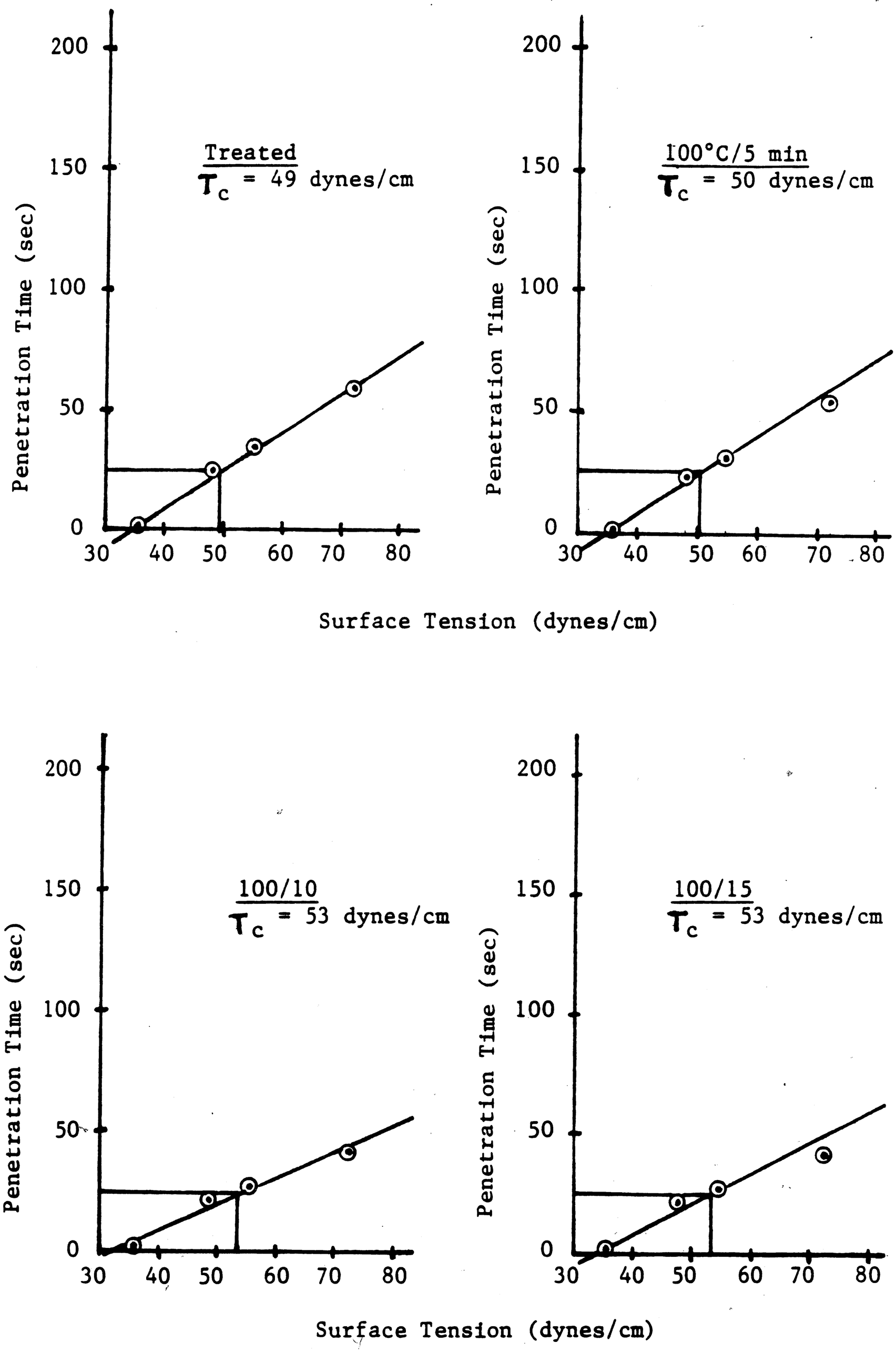


Figure 19. Plots of penetration time versus surface tension yielding the critical surface tension for several samples.

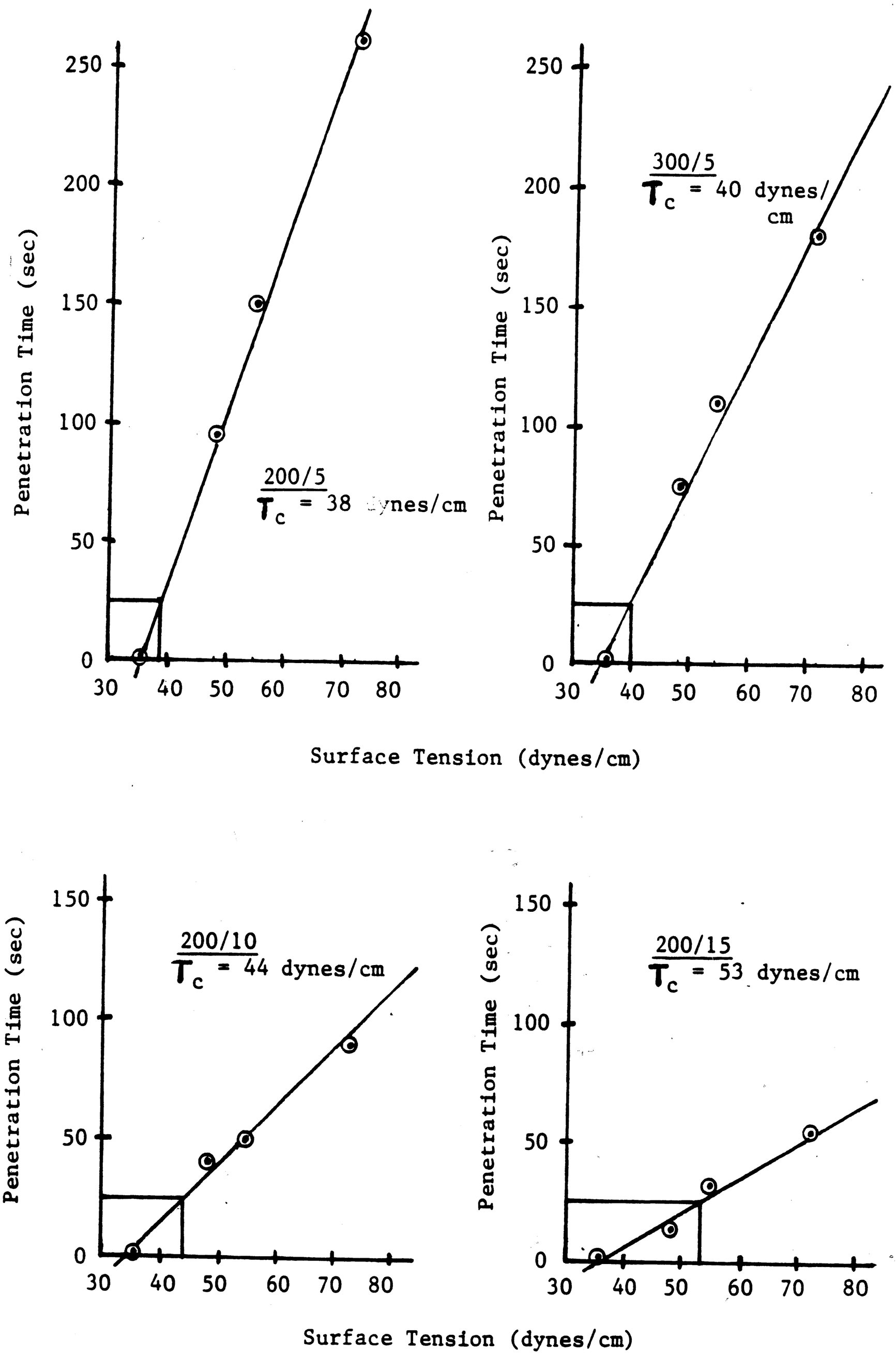


Figure 19 continued. Plots of penetration time versus surface tension yielding the critical surface tension for several samples.

Appendix E indicates that the levels are indeed similar. Furthermore, the calculations for the radius, r, also yield similar results.

After replacing the soil in the tubes with fresh, dry samples, the test was repeated using distilled, deionized water as the pore fluid. The photograph in Figure 20 shows the condition after equilibrium had been attained. The mark on each tube indicates the level to which the water rose. It is obvious that the water rose to a much greater level in the original sample. The results are tabulated in Appendix E. The calculations reveal a liquid-solid contact angle of 58.1° for the original sample. This is consistant with previous results found for clean sands (Letey et al., 1962a). The contact angles for the treated, 200/5 and 300/5 samples were found to be 74.8°, 77.9°, and 76.6° respectively. All three show a significant change with respect to the original sample. However, none are particularly close to 90° which would indicate absolute water repellency. Also, all three values are fairly close to one another, so no clear maximum can be judged by these results. By combining previous results, though, it was determined that the 200/5 sample was the most hydrophobic and could be used for permeability testing.

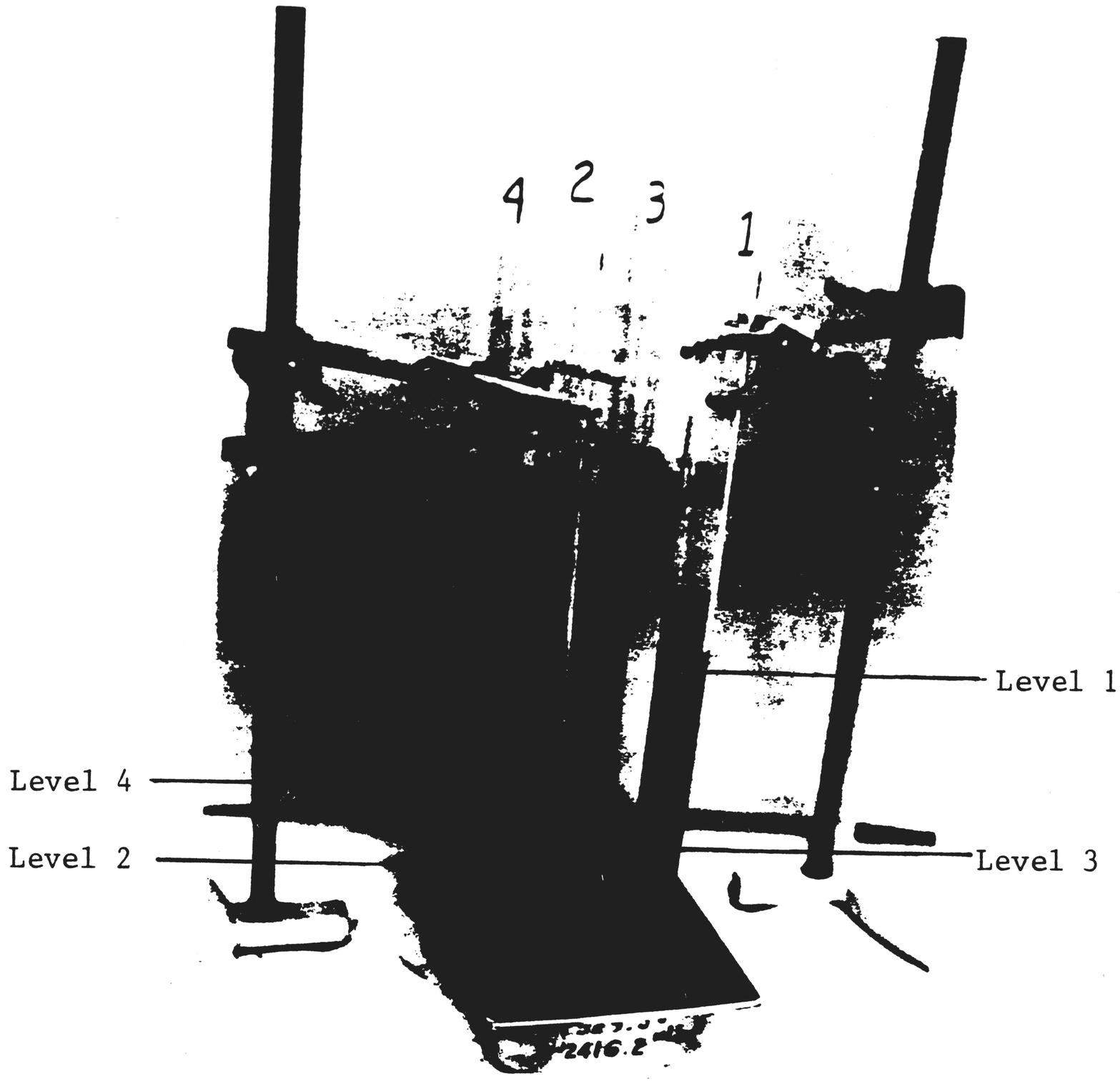


Figure 20. Capillary rise apparatus at equilibrium.

Tube Number 1: Original
Tube Number 2: Treated
Tube Number 3: 200°C/5 min
Tube Number 4: 300/5

Permeability Tests

The standard constant head permeability test was conducted on the original sample. The sand was placed dry and allowed to saturate within the apparatus. The saturation process occurred quickly (about 5 seconds for the entire column) as expected. The data and calculations in Appendix F result in a coefficient of permeability of 2.15×10^{-2} cm/sec. This is consistant with previous data for clean sands (Todd, 1980).

The 200/5 sample was tested for permeability in the Lehigh triaxial permeameter cell. The compaction procedure was found to be similar to that of the original sample in the constant head test. The molded sample in a thin latex membrane was placed between the platens within the triaxial cell. The cell was filled with water but, in an effort to reproduce the conditions found in the constant head test, only the hydrostatic cell pressure was used. Since a relatively slower rate of permeability was expected, the apparatus was set up to simulate a falling head test. The sample was permitted to saturate within the membrane with only a static head to drive the wetting front. The saturation process was much slower (about 2 minutes) than that of the original sample. Also, the 200/5 sample appeared to wet unevenly, leaving several dry pockets in the wake of the wetting front.

This was to be expected since the water had to overcome regions of particularly hydrophobic soil. Eventually, though, the entire column became wet.

Several falling head tests were run right away to determine an initial permeability rate. The data and calculations of Appendix F yield a coefficient of permeability of 4.43×10^{-4} cm/sec. This is two orders of magnitude lower than that found for the original sample. The organic coatings do, therefore, have an effect on the rate at which water moves through the soil matrix. Upon further testing, the effluent became increasingly cloudy and began to emit an odor. This indicated that the organic coating was indeed slowly dissolving and being washed out. This is assumed to result in a return to the original surface conditions of the sand particles which manifested itself in a gradual increase in permeability as testing continued.

Scanning Electron Microscope

Finally, two sets of Scanning Electron Microscope (SEM) photographs were taken of three samples: original, treated, and 200/5. The first set was taken at a magnification of 50 x (see Figure 21 A-C) and the second at 5000 x (see Figure 22 A-C). Figure 21(A) clearly shows the clean sand particles. In Figure 21(B), a dusty

coating appears on the particles. This is assumed to be the organic material. One conclusion to be drawn from this photograph is that the organic material does not coat the soil particles evenly. Figure 21(C) is quite similar to Figure 21(B), so the heating effects are not visually apparant at this level. Figure 22(A) depicts the surface of a clean sand particle as a relatively pock-marked, uneven terrain. In Figure 22(B), the coated surface appears to be much smoother. Evidently the coating has filled in the crevices to form a more uniform surface. Figure 22(C) shows a more jumbled version of the unheated surface. It is uncertain if this is a result of heating or simply an uneven coating.

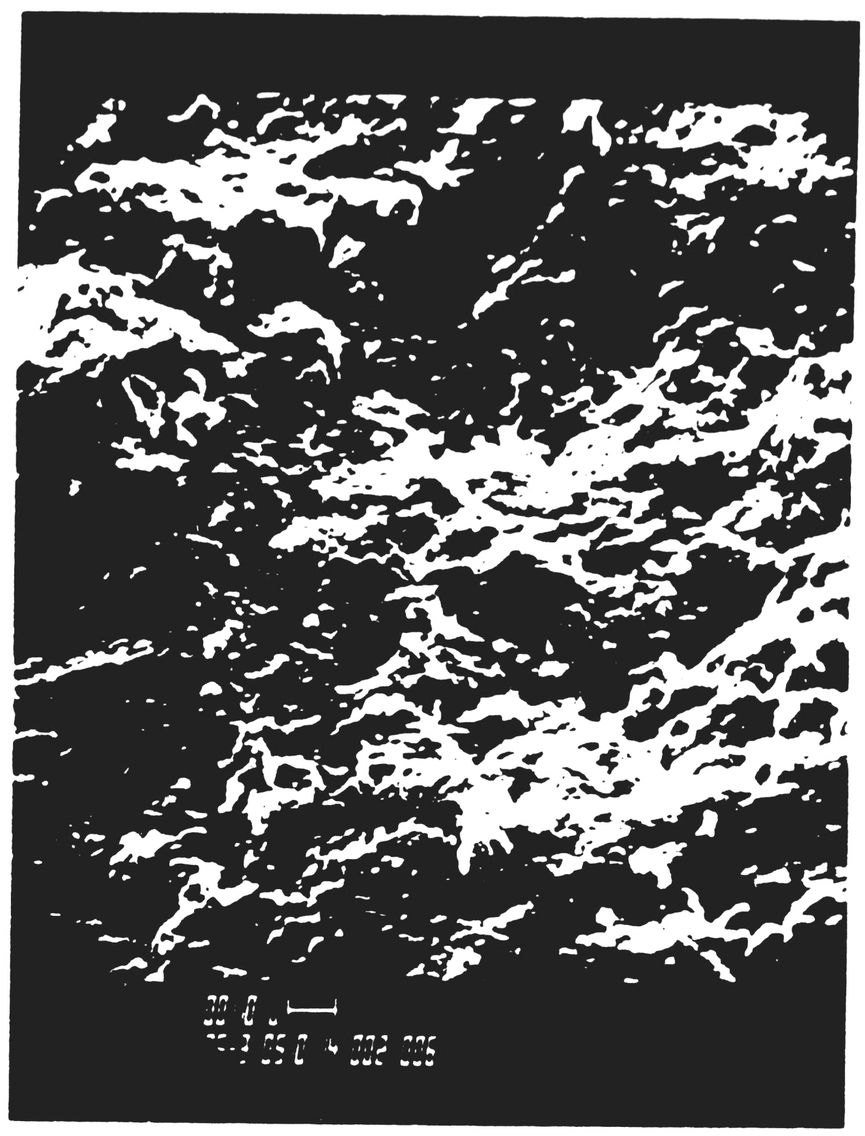
A) Original

B) Treated

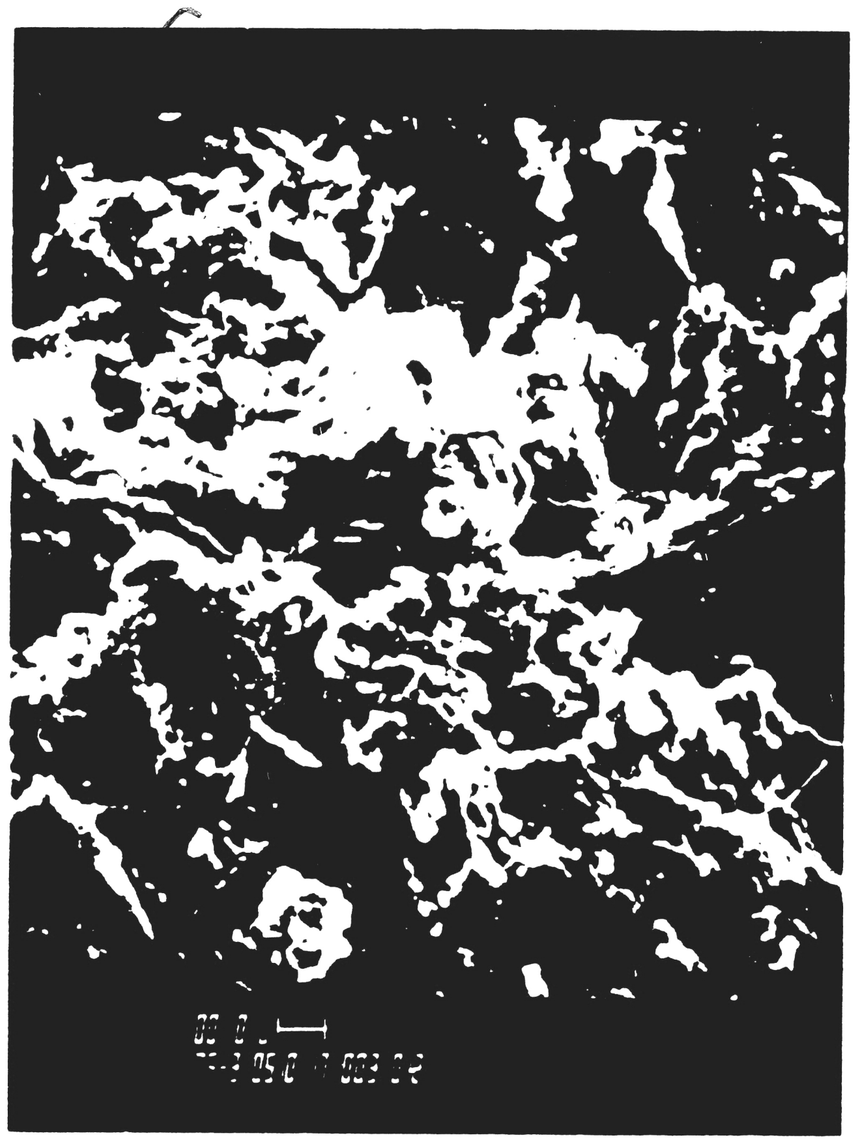
C) 200°/5 min

Figure 21. Scanning Electron Microscope (SEM) photographs of samples at 50 x magnification.

A) Original



B) Treated



C) 200°/5 min

Figure 22. Scanning Electron Microscope (SEM) photographs of samples at 5000 x magnification.

V.SUMMARY, CONCLUSION and RECOMMENDATIONS

Hydrophobic behavior is induced in sandy soils by coating the particles with a certain type of organic substance. The substance must contain a hydrophilic portion with the ability to form a metal complexation with the sand mineral, which is the mechanism by which it is adsorbed onto the particle surface. It also must contain a hydrophobic portion (usually phenolic) which will form a barrier around the sand particle. This barrier alters the surface characteristics of the particle, rendering it hydrophobic. Such an organic substance may be produced artificially, such as 4 - tertiary butylcatechol (TBC). However, naturally occurring hydrophobic soil has been found to exist. Research has isolated the cause of this phenomenon: An organic material known as a humic substance. Humic substances, specifically humic and fulvic acids, are by-products of organic decay and are synthesized by bacteria and fungi. They have the required properties and are generally considered hydrophobic. Pure extractions of humic and fulvic acids are not readily available, so an inexpensive alternative was sought.

The material chosen for this study was raw activated sludge. The theory behind this type of waste

water treatment is organic decomposition by microorganisms such as bacteria. It is likely, therefore, that humic substances exist in great quantities within the sludge material. This assumption proved to be valid as the sludge/soil mixture exhibited a marked increase in hydrophobic behavior. Also, activated sludge is a waste product that exists in great quantities, so the low-cost and high availability criteria are met.

The specific surface characteristic that renders a particle hydrophobic relates to surface tension. The affinity or repellency of a solid surface for water originates from the attractive forces between them. If the attraction between the water and the solid surface is greater than the attraction between the individual water molecules, then the water will spread out on the solid surface. However, if it is less, the solid surface will repel water. These attractive forces are the surface tensions that exist at the interfaces between solid, liquid, and vapor. This combination of forces is directly measurable as the liquid-solid contact angle. This angle, also called the wetting angle, is defined as the angle between the solid-liquid and liquid-vapor interface, or the angle that a drop of water on a solid surface makes with the plane of that surface. In other words, it is the effective angle formed by a water

meniscus in contact with the soil pore walls.

Fluid movement through soil is directly related to the liquid solid contact angle and, therefore, the surface tension characteristics of the soil and permeant. If the wetting angle is zero, there is no resistance and the fluid passes freely. This is the case for ethanol in any soil structure. Most wettable sands, though, have been found to have a wetting angle near 55^{o}. Some naturally-occuring hydrophobic soils have been found to have a wetting angle approaching or even exceeding 90^{o}. From a physical standpoint, a wetting angle of 90^{o} or greater would imply complete resistance to wetting. It is assumed, therefore, that the liquid-solid contact angle is a direct measurement of hydrophobic behavior in soils. The purpose of the organic coating on the soil particles, then, is to mask the surface tension properties of the soil and increase the wetting angle towards 90^{o}.

In reality, water can move through the soil in two ways: by saturated flow and unsaturated flow. Saturated flow occurs when all of the void spaces are occupied by water. It has traditionally been modeled by Darcy's Law and movement is in response to hydraulic gradients. Hydraulic conductivity is a factor of pore geometry, fluid viscosity, and fluid density. On the other hand,

unsaturated flow occurs when pore spaces are partially occupied by gas. Water may move either by liquid flow through the existing moisture films surrounding the soil particles or by vapor diffusion through the interconnected air spaces. Hydraulic conductivity calculations must take both into account. For the saturated condition, the wetting angle is not a flow characteristic because liquid-air interfaces are absent within the pore spaces. It is believed, therefore, that hydrophobic behavior indirectly influences the hydraulic conductivity by not permitting the soil to become fully saturated.

These concepts were verified by the results of this study. Hydraulic conductivity was decreased by two orders of magnitude in the treated soil. It was visually apparent that the soil did not wet completely at first and this directly affected the passage of water. The liquid-solid contact angle measurements also indicated a significant change in the treated soil. Clearly the organic material altered the surface tension characteristics of the soil considerably. The effects of heating upon these characteristics were examined as well. As expected, an optimum range of heating temperature and duration exists (probably between 200^{o}C and 300^{o}C for 5 minutes) which chemically changes the organic material towards maximum hydrophobic behavior. Ranges below this

optimum have little or no effect, ranges above tend to burn the organics and destroy this behavior completely.

Upon recalling that the purpose of this study was to provide an initial investigation into a low-cost alternative low-permeability material, the results are to be considered successful. The radical concept of altering the permeability of a given soil by inducing water repellency as opposed to the traditional method of physically blocking the water with fine-grained particles has been proven to be feasible. However, in light of the fact that this material is to be thought of as a replacement for clay lining materials, a great deal of improvement must be made. First of all, the coefficient of permeability must be reduced by at least three orders of magnitude to approach that of a clay liner. Secondly, the problem of solubility must be overcome. The organic coating, when dry, is an effective hydrophobic material. However, after some degree of saturation, it was observed that this effect began to diminish. It became evident that the coating was soluable in water and would wash away. Permeability, therefore, tended to increase over time. Finally, if hydrophobic soil is truly to become an improvement over clay liners, it must be equally impermeable to all permeants over a wide range of pH values. Such fluids will have an effect of the surface tension

values, so the coating must be able to withstand the worst case, or lowest surface tension.

Some specific suggestions to attain these goals are offered at this point. For one thing, the mixing and heating operations should be improved. As the SEM photographs indicate, the sand particles did not appear to be evenly coated. A method of mixing the soil with the organic material that will ensure an even, complete coating needs to be developed. The heating procedure used consisted of placing the treated soil in a porcelain bowl and simply setting it inside the furnace. It became apparent that the soil was not being evenly heated over such short durations. Perhaps a furnace with a rotating mixer can be used for this purpose.

With respect to the question of solubility, an environmental chemistry background is required to analyze ways of improving the adsorption mechanism. It is believed that some sort of catalyst may be required to ensure an insoluble bond between the soil and the organic material. It may be necessary to use a different type of organic material than what is found in activated sludge. Along these lines, the sludge itself ought to be analyzed to determine if humic substances are indeed present and in what concentrations. It may be possible to isolate the active material and mix it in higher concentrations

with the soil. Finally, a better source for humic substance may be found or an entirely different organic material such as dredged material from lake bottoms or river deltas may prove to be a better solution. Any such alternative must be inexpensive as well as plentiful to maintain the low-cost aspect of this procedure. It is hoped, therefore, that a combination of these solutions will result in a long-term, low-permeability material.

If all of these criteria are met, hydrophobic soil will certainly become an attractive alternative to clay lining materials. Since it is essentially a sand, it will not be subject to the shrinkage and cracking problems associated with clays. Also, it will be placed in the dry with minimum compaction, so construction costs should be low. An on-site treatment facility would simply be required to mix a local sandy soil with the organic coating and provide the prescribed heating procedure. Even if a very high degree of impermeability is unattainable, other more moderate applications are forseen. These include lining underground storage tank excavations, sealing well casings, and maintaining dewatered construction sites. In conclusions, then, the concept of hydrophobic soil is encouraging and certainly merits further investigation.

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APPENDIX A

ATTERBERG LIMITS DETERMINATION

Project Hydrophobic Soil Job No. CE 491

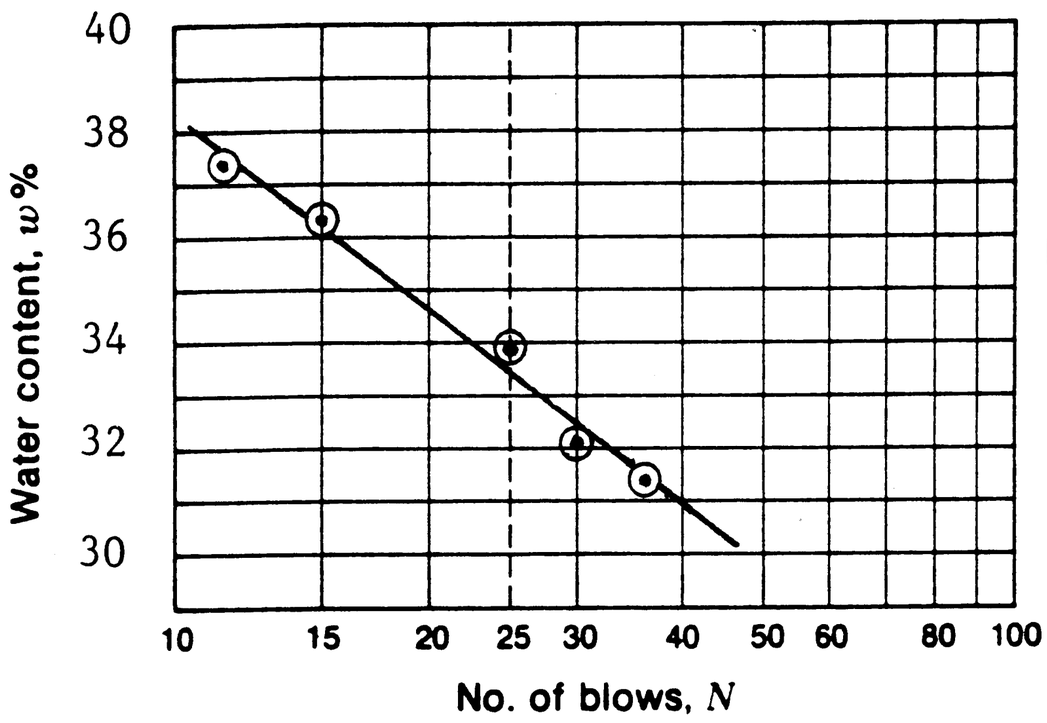
Location of Project Fritz Lab Boring No. _____ Sample No. In-situ

Description of Soil Light brown sandy clay

Depth of Sample _____ Tested By PEG Date 10/20/87

Liquid Limit Determination

Can no.	1	2	3	4	5	
Wt. of wet soil + can	28.10	26.28	25.57	25.78	26.20	
Wt. of dry soil + can	25.02	23.69	23.09	23.13	23.32	
Wt. of can	15.21	15.61	15.78	15.82	15.62	
Wt. of dry soil	9.81	8.08	7.31	7.31	7.70	
Wt. of moisture	3.08	2.59	2.48	2.65	2.88	
Water content, w%	31.4	32.1	33.9	36.3	37.4	
No. of blows, N	37	30	25	15	12	



Flow index F_i = _____

Liquid limit = 33.5

Plastic limit = 22.0

Plasticity index I_p = 11.5

Plastic Limit Determination

Can no.	1	2	3	Avg.
Wt. of wet soil + can	16.15	16.81	16.50	
Wt. of dry soil + can	16.00	16.64	16.29	
Wt. of can	15.32	15.88	15.31	
Wt. of dry soil	0.68	0.76	0.98	
Wt. of moisture	0.15	0.17	0.21	
Water content, $w\% = w_P$	22.1	22.4	21.4	22.0

GRAIN SIZE ANALYSIS–MECHANICAL

Project Hydrophobic Soil Job No. CE 491

Location of Project Fritz Lab Boring No. _____ Sample No. In-situ

Description of Soil Lt. brown sandy clay Depth of Sample _____

Tested By PEG Date of testing 10/15/87

Soil Sample Size (ASTM D1140-54)

Nominal diameter of largest particle	Approximate minimum Wt. of sample, g
No. 10 sieve	200
No. 4 sieve	500
3/4 in.	1500

Wt. of dry sample + container	946 g
Wt. of container	373 g
Wt. of dry sample, W_s	573 g

Sieve analysis and grain shape

Sieve no.	Diam. (mm)	Wt. retained	% retained	% passing
4	4.75	0	0	100.0
10	2.00	86 g	15.0	85.0
20	0.850	169	29.5	55.5
40	0.425	107	18.7	36.8
60	0.250	41	7.2	29.6
100	0.150	50	8.7	20.9
200	0.075	26	4.5	16.4
PAN	-	94	16.4	0.0
		Σ = 573	Σ = 100.0	

% passing = 100 − $\sum$ % retained.

GRAIN SIZE ANALYSIS-HYDROMETER METHOD

Project Hydrophobic Soil Job No. CE 491

Location of Project Fritz Lab Boring No. _____ Sample No. In-situ

Description of Soil Light brown silty clay Depth of Sample _____

Tested By PEG Date of Testing 10/22/87

Hydrometer analysis

Hydrometer no. 152H G_s of solids = 2.70 a = 0.99

Dispersing agent $NaPO_3$ (Calgon) Amount 4% in 115 ml Wt. of soil, W_s, 573 g

Zero correction +5 Meniscus correction 1

Date	Time of reading	Elapsed time, min	Temp., °C	Actual Hyd. reading R_a	Corr. Hyd. reading R_c	% Finer	Hyd. Corr. only for meniscus, R	L from Table 6-5	$\frac{L}{t}$	K from Table 6-4	D, mm
		1	25	30.1	26.4	4.56	31.1	11.2	11.2	0.0127	0.0425
		2	25	30.0	26.3	4.54	31.0	11.2	5.60		0.0300
		3	25	29.8	26.2	4,53	30.8	11.2	3.73		0.0245
		4	25	29.6	26.0	4.49	30.6	11.3	2.825		0.0213
		8	25	29.0	25.4	4.39	30.0	11.4	1.425		0.0152
		15	25	29.1	25.5	4.41	30.1	11.4	0.760		0.0111
		30	24	26.5	22.5	3.89	27.5	11.8	0.393	.0128	0.0080
		60	24	25.3	21.3	3.68	26.3	12.0	0.200		0.0057
		120	24	23.7	19.7	3.40	24.7	12.3	.1025		0.0041
		240	24	21.8	17.8	3.08	22.8	12.5	.0521		0.0029
		480	23	20.5	16.2	2.80	21.5	12.8	.0267	.0130	0.0021
		1380	23	20.0	15.7	2.71	21.0	12.9	.0093	.0130	0.0013
		1800	24	19.3	15.3	2.64	20.3	13.0	.0072	.0128	0.0011
		$(C_t)_{25}$	= +	1.30							
		$(C_t)_{24}$	= +	1.00							
		$(C_t)_{23}$	= +	0.70							

$R_c = R_{actual}$ − zero correction + C_T % finer = $R_c(a)/W_s$ $D = K\sqrt{L/t}$

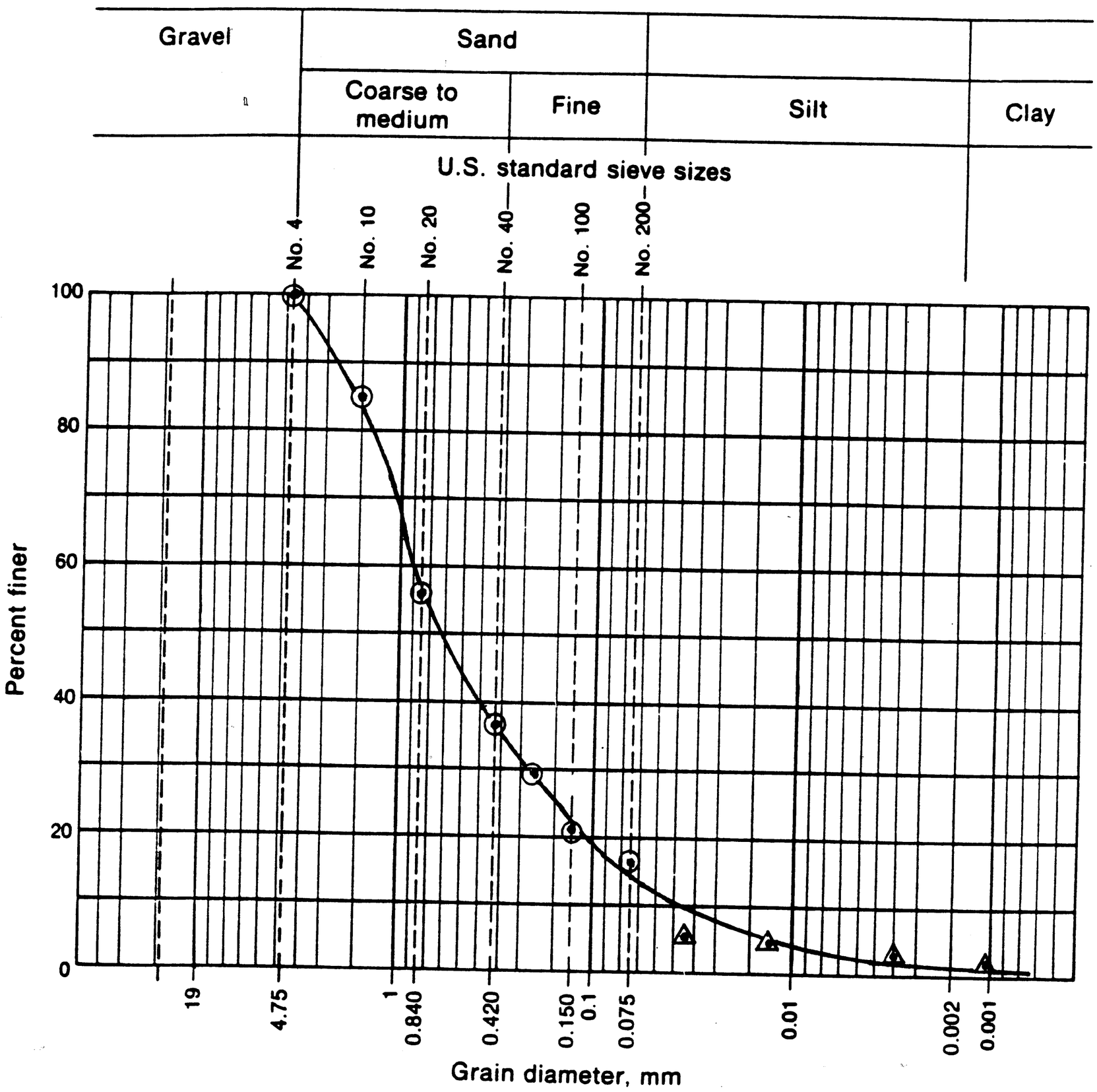
GRAIN SIZE DISTRIBUTION

Project Hydrophobic Soil Job. No. CE 491

Location of Project Fritz Lab Boring No. _____ Sample No. In-situ

Description of Soil Lt. brown sandy clay Depth of Sample _____

Tested By. PEG Date of Testing 10/15/87



Visual soil description Light brown sandy clay

Soil classification:

SC System USC

GRAIN SIZE ANALYSIS–MECHANICAL

Project Hydrophobic Soil Job No. CE 491

Location of Project Fritz Lab Boring No. _____ Sample No. Washed

Description of Soil Lt. brown sandy soil Depth of Sample _____

Tested By PEG Date of testing 3/7/88

Soil Sample Size (ASTM D1140-54)

Nominal diameter of largest particle	Approximate minimum Wt. of sample, g
No. 10 sieve	200
No. 4 sieve	500
3/4 in.	1500

Wt. of dry sample + container	1380 g
Wt. of container	272 g
Wt. of dry sample, W_s	1108 g

Sieve analysis and grain shape

Sieve no.	Diam. (mm)	Wt. retained	% retained	% passing
4	4.75	0	0	100.0
10	2.00	175	15.8	84.2
30	0.600	398	35.9	48.3
50	0.300	242	21.9	26.4
100	0.150	235	21.2	5.2
140	0.106	37	3.3	1.9
PAN	-	21	1.9	0.0
		Σ = 1108	Σ = 100.0	

% passing = 100 − $\sum$ % retained.

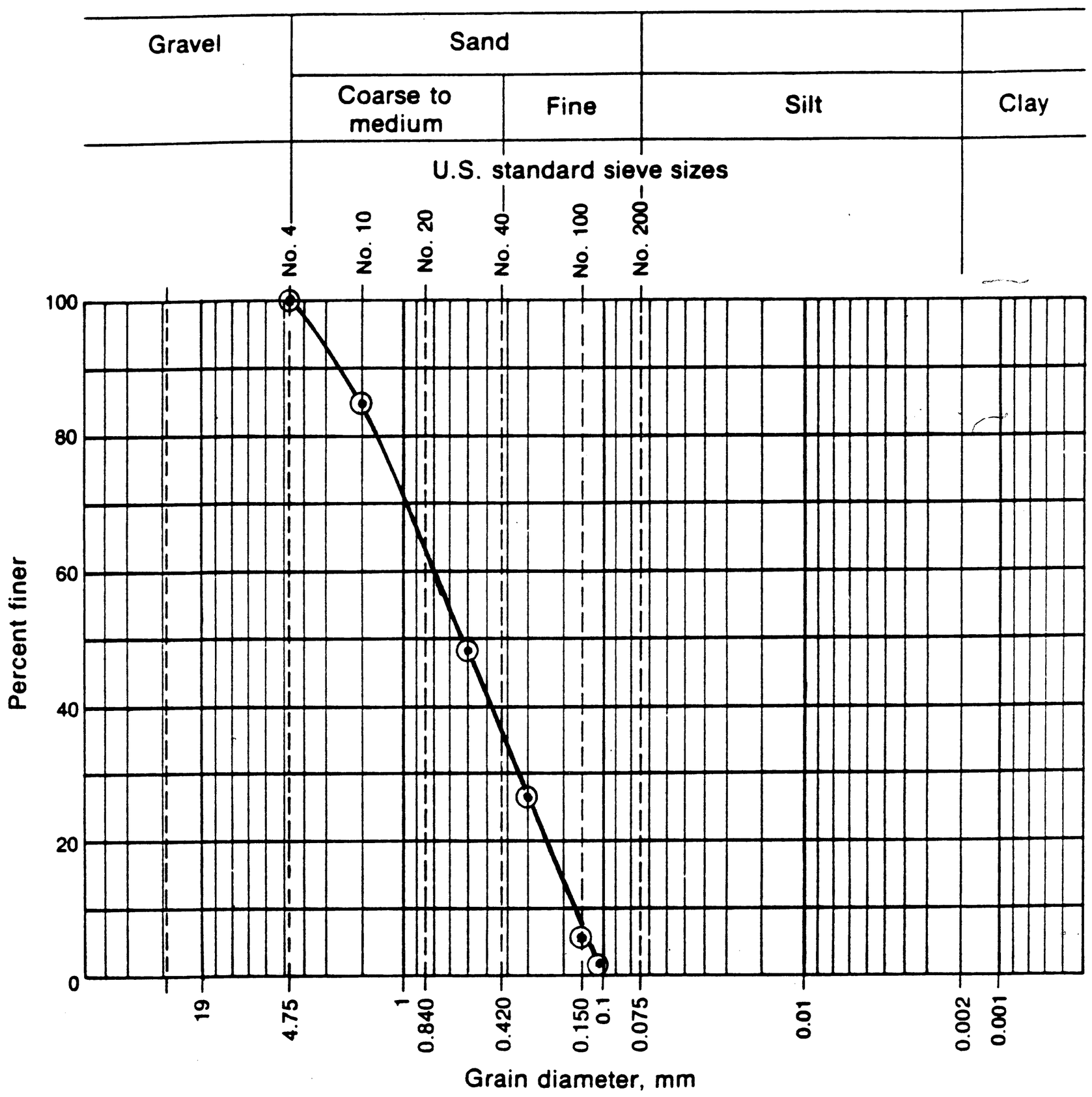
GRAIN SIZE DISTRIBUTION

Project Hydrophobic Soil Job. No. CE 491

Location of Project Fritz Lab Boring No. _____ Sample No. Washed

Description of Soil Lt. brown sandy soil Depth of Sample _____

Tested By. PEG Date of Testing 3/7/88



Visual soil description Light brown sandy soil

Soil classification:

SP System USC

APPENDIX B

SANDY SOIL WITH SLUDGE

1) Weight Pan	167 g
2) Weight Pan + Soil	771 g
3) Weight Pan + Soil + Sludge (wet)	1132 g
4) Weight Pan + Soil + Sludge (dry)	784 g
5) Weight Soil (2 - 1)	604 g
6) Weight Sludge (wet) (3 - 2)	361 g
7) Weight Sludge (dry) (4 - 2)	13 g
8) Water Content $\frac{(6 - 7)}{(5 + 7)} \times 100\%$	56.4 %
9) Percent Solid Added $(\frac{7}{5} \times 100\%)$	2.15 %

APPENDIX C

WATER DROP TEST

Sample	Average WDPT[1] (sec)
Original	0
Treated	90
100°C/5 min	80
100/10	70
100/15	67
200/5	540
200/10	300
200/15	85
300/5	420
300/10	0
300/15	0

1. Water Drop Penetration Time

APPENDIX D

CRITICAL SURFACE TENSION TEST

	Average Drop Penetration Time (sec)						
	% By Volume of Ethyl Alcohol in Water @ 40°C				Distilled Deionized Water	% By Weight of NaOH in Water @ 18°C	
Percent	48.0	24.0	10.0	5.0		16.66	35.90
Surface Tension (dynes/cm)	28.93	35.50	48.25	54.92	72.10	83.05	101.05
Samples: Original				-	0	-	0.5
Treated	-	1.5	25	34	60	-	
100°C/5 min	-	1.3	24	32	56	-	
100/10	-	1.2	22	27	42	-	
100/15	-	1.0	23	27	40	-	
200/5	-	1.5	95	150	260	-	
200/10	-	1.0	40	50	90	-	
200/15	-	0.5	14	32	55	-	
300/5	-	1.3	75	110	180	-	
300/10				-	0	-	0.5
300/15				-	0	-	0.5

APPENDIX E

LIQUID - SOLID CONTACT ANGLE TEST

Sample	Height of Capillary Rise (cm)	
	Capillary Fluid	
	Ethanol	Water
Original	15.0	20.5
Treated	13.3	9.0
200°C/5 min	11.8	6.4
300/5	13.5	8.1

Surface Tensions (γ):

Ethanol @ 20°C: γ_E = 22 dynes/cm

Distilled, Deionized Water @ 20°C: γ_W = 72.1 dynes/cm

Densities (ρ):

Ethanol: ρ_E = 0.7893 g/cm^3

Water: ρ_W = 1.0 g/cm^3

Calculations:

Solve for r:

$$r = \frac{2\gamma_E}{\rho_E g h_E} \qquad \text{(Eq. 6)}$$

where $g = 980.7\ cm/s^2$

$$r_{orig} = \frac{2\ (22\ g\ cm/s^2)/cm}{0.7893\ g/cm^3\ (980.7\ cm/s^2)(15.0\ cm)}$$

$$= 3.79 \times 10^{-3}\ cm$$

Sample	Original	Treated	200/5	300/5
r (cm)	3.79×10^{-3}	4.27×10^{-3}	4.82×10^{-3}	4.21×10^{-3}

Solve for θ:

$$\cos\theta = \frac{h_W \rho_W g r}{2\gamma_W} \qquad \text{(Eq. 7)}$$

$$(\cos\theta)_{orig} = \frac{(20.5\ cm)(1.0\ g/cm^3)(980.7\ cm/s^2)(3.79 \times 10^{-3} cm)}{2\ (72.1\ g\ cm/s^2)/cm}$$

$$= 0.528$$

$$\theta = 58.1°$$

Sample	Original	Treated	200/5	300/5
θ	58.1°	74.8°	77.9°	76.6°

APPENDIX F

COEFFICIENT OF PERMEABILITY (Constant Head)

Project Hydrophobic Soil Job No. CE 491

Location of Project Fritz Lab

Description of Soil Light Brown sandy soil (original)

Tested by PEG Date of Testing 5/10/88

Sample Dimensions: Diam. 6.3 cm; Area 31.17 cm²; Ht. 13.0 cm

Vol. 405.2 cm³

h = 85.0 cm

Test data *Test data used*

Test No.	*t*, s	Q, cm³	*T*, °C	Test No.	*t*, s	Q, cm³	*T*, °C
1	120	650	25.0				
2	180	780	21.7				
3	180	750	18.6				
4	180	720	17.8				
				Average			

Calculations:

$$k_T = \frac{Q\ L}{A\ h\ t} \qquad \text{(Eq. 8)}$$

$$(k_T)_1 = \frac{650\ cm^3\ (13\ cm)}{(31.17\ cm^2)(85.0\ cm)(120\ s)}$$

$$= 2.66 \times 10^{-2}\ cm/s$$

$$\frac{(\eta_T)_1}{\eta_{20}} = 0.8893 \qquad \text{(From Table F - 1)}$$

$$(k_{20})_1 = 0.8893(2.66 \times 10^{-2}) = 2.36 \times 10^{-2}\ cm/s$$

Trial	1	2	3	4
k_T (cm/s)	2.66×10^{-2}	2.13×10^{-2}	2.04×10^{-2}	1.96×10^{-2}
η_T/η_{20}	0.8893	0.9600	1.0351	1.0560
k_{20} (cm/s)	2.36×10^{-2}	2.04×10^{-2}	2.12×10^{-2}	2.07×10^{-2}

Average $k_{20} = 2.15 \times 10^{-2}$ cm/s

COEFFICIENT OF PERMEABILITY (Falling Head)

Project Hydrophobic Soil Job No. CE 491

Location of Project Fritz Lab

Description of Soil Dark brown sandy soil (200/5)

Tested by A. Sebuktekin Date of Testing 5/5/88

Sample Dimensions: Diam. 14.0 cm; Area 153.9 cm²; Ht. 7.34 cm

Vol. 1130 cm³

Standpipe = [burette, other (specify)] Teflon® tube

Area of standpipe, a = 0.34 cm²

Test data *Test data used*

Test no.	h_1, cm	h_2, cm	t, s	Q_{in}, cm³	Q_{out}, cm³	T, °C	Test no.	h_1, cm	h_2, cm	t, s	T, °C
1	87.3	14.5	65	–	–	20	5	87.2	18.0	60	20
2	87.0	14.1	65	–	–	20	6	87.2	17.0	60	20
3	87.2	16.3	60	–	–	20	7	87.2	15.0	66	20
4	87.2	17.0	60	–	–	20					
							Average	87.2	16.0	62	20

Calculations:

$$k_T = \frac{a\ L}{A\ t} \ln (h_1/h_2) \qquad \text{(Eq. 9)}$$

$$= \frac{0.34\ \text{cm}^2\ (7.34\ \text{cm})}{153.9\ \text{cm}^2\ (62\ \text{s})} \ln (87.2/16.0)$$

$$= 4.43 \times 10^{-4}\ \text{cm/s}$$

$$\frac{\eta_T}{\eta_{20}} = 1.0$$

$$k_{20} = 4.43 \times 10^{-4}\ \text{cm/s}$$

Table F - 1. Viscosity corrections for η_T/η_{20}.

°C	0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
10	1.3012	1.2976	1.2940	1.2903	1.2867	1.2831	1.2795	1.2759	1.2722	1.2686
11	1.2650	1.2615	1.2580	1.2545	1.2510	1.2476	1.2441	1.2406	1.2371	1.2336
12	1.2301	1.2268	1.2234	1.2201	1.2168	1.2135	1.2101	1.2068	1.2035	1.2001
13	1.1968	1.1936	1.1905	1.1873	1.1841	1.1810	1.1777	1.1746	1.1714	1.1683
14	1.1651	1.1621	1.1590	1.1560	1.1529	1.1499	1.1469	1.1438	1.1408	1.1377
15	1.1347	1.1318	1.1289	1.1260	1.1231	1.1202	1.1172	1.1143	1.1114	1.1085
16	1.1056	1.1028	1.0999	1.0971	1.0943	1.0915	1.0887	1.0859	1.0803	1.0802
17	1.0774	1.0747	1.0720	1.0693	1.0667	1.0640	1.0613	1.0586	1.0560	1.0533
18	1.0507	1.0480	1.0454	1.0429	1.0403	1.0377	1.0351	1.0325	1.0300	1.0274
19	1.0248	1.0223	1.0198	1.0174	1.0149	1.0124	1.0099	1.0074	1.0050	1.0025
20	1.0000	0.9976	0.9952	0.9928	0.9904	0.9881	0.9857	0.9833	0.9809	0.9785
21	0.9761	0.9738	0.9715	0.9692	0.9669	0.9646	0.9623	0.9600	0.9577	0.9554
22	0.9531	0.9509	0.9487	0.9465	0.9443	0.9421	0.9399	0.9377	0.9355	0.9333
23	0.9311	0.9290	0.9268	0.9247	0.9225	0.9204	0.9183	0.9161	0.9140	0.9118
24	0.9097	0.9077	0.9056	0.9036	0.9015	0.8995	0.8975	0.8954	0.8934	0.9813
25	0.8893	0.8873	0.8853	0.8833	0.8813	0.8794	0.8774	0.8754	0.8734	0.8714
26	0.8694	0.8675	0.8656	0.8636	0.8617	0.8598	0.8579	0.8560	0.8540	0.8521
27	0.8502	0.8484	0.8465	0.8447	0.8428	0.8410	0.8392	0.8373	0.8355	0.8336
28	0.8318	0.8300	0.8282	0.8264	0.8246	0.8229	0.8211	0.8193	0.8175	0.8157
29	0.8139	0.8122	0.8105	0.8087	0.8070	0.8053	0.8036	0.8019	0.8001	0.7984
30	0.7967	0.7950	0.7934	0.7917	0.7901	0.7884	0.7867	0.7851	0.7834	0.7818
31	0.7801	0.7785	0.7769	0.7753	0.7737	0.7721	0.7705	0.7689	0.7673	0.7657
32	0.7641	0.7626	0.7610	0.7595	0.7579	0.7564	0.7548	0.7533	0.7517	0.7502
33	0.7486	0.7471	0.7456	0.7440	0.7425	0.7410	0.7395	0.7380	0.7364	0.7349
34	0.7334	0.7320	0.7305	0.7291	0.7276	0.7262	0.7247	0.7233	0.7218	0.7204
35	0.7189	0.7175	0.7161	0.7147	0.7133	0.7120	0.7106	0.7092	0.7078	0.7064

VITA

The author was born on October 22, 1962 in Red Bank, NJ. The son of Barry and Ruth Gauffreau and older brother of Stuart Gauffreau, he grew up in Middletown, NJ along the Jersey Shore. He graduated from Christian Brothers Academy, Lincroft, NJ in 1980. He entered Lehigh University in the fall of 1980 as a Freshman engineer. While at Lehigh, he became an active member of Psi Upsilon Fraternity, serving as its Steward and Rush Chairman. At present, he is a member of the Board of Trustees of the Goodale Literary Association, the Eta Chapter of Psi Upsilon's alumni organization. Also as an undergraduate, he was elected to membership in the Chi Epsilon and Tau Beta Pi honor societies. He graduated with honors from Lehigh in June, 1984 with a BS in Civil Engineering.

Upon graduation, the author was employed as a designer in the Civil/Site department of Lockwood Greene Engineers, Inc. in New York City. While there, he became the department CADD Coordinator and a "Job Captain" responsible for the site design of several major construction projects. In August of 1986, he returned to Lehigh University to persue his Master's Degree in Civil Engineering, specializing in Geotechnical Engineering. During

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